INTERNATIONAL STANDARD

ISO 16859-3

First edition
2015-09-15

Metallic materials — Leeb hardness test —

Part 3: Calibration of reference test blocks

Matériaux métalliques — Essai de dureté Leeb —

Partie 3: Etalonnage des blocs de référence

Reference number
ISO 16859-3:2015(E)

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Foreword

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The committee responsible for this document is ISO/TC 164, *Mechanical testing of metals*, Subcommittee SC 3, *Hardness testing*.

ISO 16859 consists of the following parts, under the general title *Metallic materials — Leeb hardness test*:

— *Part 1: Test method*

— *Part 2: Verification and calibration of the testing devices*

— *Part 3: Calibration of reference test blocks*

Metallic materials — Leeb hardness test —

Part 3:
Calibration of reference test blocks

1 Scope

This part of ISO 16859 specifies a method for the calibration of reference test blocks that are used for the indirect verification of Leeb hardness testers according to ISO 16859-2 and for the periodic checking according to ISO 16859-1.

The procedures necessary to ensure metrological traceability of the calibration machine are also specified.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 16859-1, *Metallic materials — Leeb hardness test — Part 1: Test method*

ISO 16859-2, *Metallic materials — Leeb hardness test — Part 2: Verification and calibration of the testing devices*

ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories*

3 Manufacture of reference test blocks

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

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

3.2 The uniformity of the metallic reference test block shall meet the requirements specified in 7.2 and Table 3.

3.3 The impact nature of a Leeb test requires a reference test block with a minimum mass and thickness, as specified in Table 1.

NOTE Examples of common dimensions of reference test blocks are specified in Annex C.

Table 1 — Mass and thickness requirements of reference test block

Type of impact devices	Minimum thickness mm	Minimum diameter mm	Minimum mass kg
D, DL, D+15, S, E, C	33	85	2,7
G	65	115	6,0

3.4 The reference test blocks shall be free of magnetism prior to calibration.

3.5 The maximum deviation from surface flatness of the top and bottom surfaces shall not exceed 0,01 mm. The surfaces of the blocks shall not be convex.

The maximum deviation from parallelism of the top and bottom surfaces shall not exceed 0,02 mm per 50 mm.

3.6 The test surfaces shall be free from damage, such as notches, scratches, oxide layers, etc., which interfere with the mechanics of the indentation process.

The mean surface roughness, R_a, [2] of the test surface(s) shall not exceed 0,1 µm. The sampling length, *l*, is 0,80 mm (see ISO 4287:1997, 3.1.9).

3.7 To provide evidence that no material is removed from the test surface(s) of the reference test block subsequent to calibration, the thickness at the time of calibration shall be marked on the test surface(s) or printed on the calibration certificate to an accuracy of 0,1 mm. Alternatively, a mark shall be placed on both the top and bottom surfaces, see 8.1 e).

4 Calibration machine

4.1 General

In addition to fulfilling the general conditions specified in ISO 16859-2:2015, Clause 3, the calibration machine shall also meet the requirements given in 4.2 to 4.4.

4.2 Traceability

4.2.1 Leeb primary hardness standard machines owned by national level institutions are used to calibrate primary reference test blocks in Leeb hardness for accredited Leeb hardness calibration laboratories according to ISO/IEC 17025.

4.2.2 The instruments used for the verification and calibration of the Leeb hardness calibration machine shall be traceable to national standards.

NOTE A four-level structure of the metrological chain is necessary to define and disseminate hardness scales. The metrological chain of hardness values obtained by the Leeb method is defined in ISO 16859-1:2015, Figure C.1.

4.3 Requirements on calibration machines

4.3.1 Requirements on Leeb hardness calibration machines are given in Annex A.

4.3.2 The resolution of Leeb hardness calibration machines shall be equal to or better than 1,0 HL.

4.4 Calibration of calibration machines

4.4.1 Leeb hardness calibration machines shall be calibrated at an interval of <12 months.

4.4.2 Leeb hardness calibration machines shall comply with the requirements defined in Annex A.

4.4.3 Following direct calibration, an indirect calibration shall be conducted with at least three primary reference test blocks that cover the complete range of the Leeb hardness scale, as defined in Table 2.

Table 2 — Leeb hardness ranges, minimum repeatability, and limiting error of calibration machines

Type of impact device	Leeb hardness range for indirect calibration HL[a]	Minimum repeatability W_H %	Limiting error G_H %
D, D+15	<500	1,0	±2,0
	500 to 700	1,0	±1,5
	>700	1,0	±1,0
DL, S	<700	1,0	±2,0
	700 to 850	1,0	±1,5
	>850	1,0	±1,0
C, E	<600	1,0	±2,0
	600 to 750	1,0	±1,5
	>750	1,0	±1,0
G	<450	1,0	±2,0
	450 to 600	1,0	±1,5
	>600	1,0	±1,0

[a] HLD for impact devices D, HLD+15 for impact devices D+15, HLDL for impact devices DL, HLS for impact devices S, HLC for impact devices C, HLE for impact devices E, HLG for impact devices G.

Indirect calibration comprises at least 10 readings on each reference test block.

4.4.4 Calculation of error and repeatability of indirect calibration:

$$\overline{H} = \frac{1}{n}\sum_{1}^{n} H_{i} \quad (1)$$

where

$\overline{H}$ is the Leeb hardness mean value;

H_i is the single Leeb hardness reading.

$$b_H = \frac{1}{n}\sum_{1}^{n} H_{i} - H_{CRM} \quad (2)$$

where

H_{CRM} is the Leeb hardness of primary reference test block;

b_H is the error of Leeb hardness.

In indirect calibration, requirements concerning the limiting error of Leeb hardness are met when

$$G_H \geq |b_H(H)| + u_{CRM} \tag{3}$$

where

G_H is the limiting error of Leeb hardness (see Table 2);

u_{CRM} is the calibration uncertainty of primary reference test blocks according to the calibration certificate for $k = 1$.

Standard deviation, s_H

$$s_H = \sqrt{\frac{\sum_1^n (H_i - \overline{H})^2}{n-1}} \tag{4}$$

Variation coefficient, V_H

$$V_H = \frac{s_H}{\overline{H}} \cdot 100\,\% \tag{5}$$

In indirect calibration, requirement concerning the minimum repeatability, W_H, (see Table 2) of Leeb hardness are met when:

$$W_H \geq V_H \tag{6}$$

where W_H is the minimum repeatability of Leeb hardness (see Table 2).

5 Calibration procedure

5.1 Reference test blocks are calibrated at a temperature of (23 ± 5) °C using Leeb hardness calibration machines conforming to Clause 4, using the general procedure described in ISO 16859-1.

During calibration, thermal drift shall not exceed 1 °C.

5.2 Reference test blocks are placed on a rigid steel plate of minimum thickness of 45 mm and minimum mass of 45 kg, and whose contact surface has been ground to a flatness of 0,01 mm or better. The reference test block is coupled to steel plate using a thin plastic film (thickness <0,1 mm). The plastic film shall serve for adhesion between the block and the steel plate.

6 Number of test indentations

Leeb reference test blocks can be calibrated on both block surfaces (side A and side B). During a calibration sequence, 10 indentations are made evenly distributed over the entire test surface for each side. The calibration value is calculated as the arithmetic mean of 10 single readings. The calibration value is assigned to the respective test surface.

7 Uniformity of hardness

7.1 The Leeb hardness values of a test surface A of a reference test block are denoted H_1, H_2, ..., H_{10}. If the block has two test surfaces, the Leeb hardness values of test surface B are denoted H_{11}, H_{12}, ..., H_{20}. The arithmetic mean value(s) from the Leeb hardness calibration $\overline{H}_A$ (and $\overline{H}_B$) are calculated using Formula (7) and Formula (8).

$$\overline{H}_A = \frac{H_1 + H_2 + ... + H_{10}}{10} \quad (7)$$

$$\overline{H}_B = \frac{H_{11} + H_{12} + ... + H_{20}}{10} \quad (8)$$

7.2 The variation coefficient provides the statistical parameter for the dispersion of the calibration values. Validity of results for the variation coefficients is defined in Table 3.

Standard deviation:

$$s_{H_A} = \sqrt{\frac{\sum_{1}^{10} (H_i - \overline{H}_A)^2}{n-1}} \quad (9)$$

$$s_{H_B} = \sqrt{\frac{\sum_{11}^{20} (H_i - \overline{H}_B)^2}{n-1}} \quad (10)$$

Variation coefficient:

$$V_{H_A} = \frac{s_{H_A}}{\overline{H}_A} \cdot 100 \quad \text{in } \% \quad (11)$$

$$V_{H_B} = \frac{s_{H_B}}{\overline{H}_B} \cdot 100 \quad \text{in } \% \quad (12)$$

Table 3 — Maximum variation coefficient for reference test block calibrations

Type of impact device	Leeb hardness range of reference test block HL[a]	Maximum variation coefficient %
D, D+15	<500	2,0
	500 to 700	1,5
	>700	1,0
DL, S	<700	2,0
	700 to 850	1,5
	>850	1,0
C, E	<600	2,0
	600 to 750	1,5
	>750	1,0
G	<450	2,0
	450 to 600	1,5
	>600	1,0

[a] HLD for impact devices D, HLD+15 for impact devices D+15, HLDL for impact devices DL, HLS for impact devices S, HLC for impact devices C, HLE for impact devices E, HLG for impact devices G.

7.3 Estimation of measurement uncertainty for reference test blocks is given in Annex B.

8 Marking

8.1 Reference test blocks shall at least hold the following marks (preferably in form of an engraving on the edge of the test surface):

a) arithmetic mean value from Leeb hardness calibration incl. specification of device type, e.g. 799 HLD;

b) name or mark of manufacturer or supplier of reference test block;

c) serial number;

d) name or mark of calibration agency;

e) thickness of reference test block or markings on both test surfaces, respectively (see 3.7);

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

8.2 The mark for test surface A (B) that is put on the side of the block shall be upright when the test surface A (B) is the upper face.

8.3 Reference test blocks shall hold accompanying documentation (a calibration certificate) providing the following information:

a) a reference to this part of ISO 16859 , i.e. ISO 16859-3;

b) identification of reference test block;

c) single hardness values, arithmetic means and variation coefficients of reference test block;

d) measurement uncertainty of calibration;

e) date of calibration/date of issue.

9 Validity

Validity of a reference test block is limited to the testing conditions and for the impact device type used during calibration.

Validity of the calibration should be limited to five years. Attention is drawn to the fact that, for Al- and Cu-alloys, the calibration validity should be reduced to two to three years.

Annex A
(normative)

Requirements for Leeb hardness calibration machines

A.1 Leeb hardness calibration machines according to the principle of velocity measurement

Table A.1 gives the requirements for Leeb hardness calibration machines.

Table A.1 — Requirements on parameter implementation for Leeb hardness calibration machines

Parameter	Unit	Type of impact device						
		D	S	E	DL	D+15	C	G
Impact velocity, v_A	m/s	2,05	2,05	2,05	1,82	1,7	1,4	3,0
Maximum limiting deviation of impact velocity	m/s	±0,0025	±0,0025	±0,0025	±0,002	±0,002	±0,002	±0,005
Mass of impact body, *m*	g	5,45 ± 0,03	5,40 ± 0,03	5,45 ± 0,03	7,25 ± 0,03	7,75 ± 0,03	3,1 ± 0,03	20,0 ± 0,03
Radius of indenter ball, *r*	mm	1,5 ± 0,003	1,5 ± 0,003	1,5 ± 0,003	1,39 ± 0,003	1,5 ± 0,003	1,5 ± 0,003	2,5 ± 0,003
Material of indenter		WC-Co[a]	C[b]	PCD[c]	WC-Co[a]	WC-Co[a]	WC-Co[a]	WC-Co[a]
Vickers hardness of indenter	HV2	1600 ± 50	1600 ± 50	≥ 4500	1600 ± 50	1600 ± 50	1600 ± 50	1600 ± 50

[a] Tungsten-carbide cobalt.

[b] Ceramics.

[c] Polycrystalline diamond.

Annex B
(informative)

Measurement uncertainty of reference test block

NOTE The structure of the metrological chain that is required to define and reproduce hardness scales is shown in ISO 16859-1:2015, Figure C.1.

B.1 Direct calibration and verification of Leeb hardness calibration machines

B.1.1 Calibration and verification of geometrical parameters of impact body and support ring

See ISO 16859-2:2015, 4.3.

B.1.2 Calibration and verification of signal of electronic unit

See ISO 16859-2:2015, 4.4.

B.2 Indirect calibration and verification of Leeb hardness calibration machines

NOTE In this Annex, "reference test block" is abbreviated as "certified reference material (CRM)".

The overall performance of Leeb hardness calibration machines is verified by indirect verification using primary reference test blocks. Also, this is used to determine the repeatability and error of the Leeb hardness calibration machine from the actual Leeb hardness.

The measurement uncertainty for the indirect verification[3] of Leeb hardness calibration machine is calculated according to Formula (B.1):

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

where

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

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

u_{CRM-D} is the standard uncertainty due to the change of Leeb hardness of primary reference test blocks since the last calibration, due to drift;

u_{ms} is the standard uncertainty due to resolution of measuring system.

EXAMPLE See Tables B.1, B.2, B.3, and B.4.

primary reference test block:	(767 ± 5,0) HLD
uncertainty of measurement of primary reference test block:	u_{CRM-P} = 2,5 HLD
temporal drift of primary reference test block:	u_{CRM-D} = 0
resolution of measurement system:	δ_{ms} = 1 HLD

Table B.1 — Results of indirect calibration

Nr	**Displayed Leeb hardness value** HLD
1	763
2	768
3	767
4	768
5	764
6	767
7	765
8	767
9	770
10	769
Mean value	766,8
Standard deviation $s_{\times CRM\text{-}1}$	2,2
Standard measurement uncertainty $u_{\times CRM\text{-}1}$	0,74

$$u_{\times CRM-1} = \frac{t \cdot s_{\times CRM-1}}{\sqrt{n}} \quad \text{(B.2)}$$

with Student's t-factor t = 1,06 for n = 10:

$u_{\times CRM-1}$ = 0,74 HLD

Table B.2 — Components of measurement uncertainty

Quantity X_i	**Estimated value** x_i HLD	**Standard measurement uncertainty** $u(x_i)$ HLD	**Type of distribution**	**Sensitivity coefficient** c_i	**Contribution to uncertainty** $u_i(H)$ HLD
$u_{CRM\text{-}P}$	767	2,5	Normal	1,0	2,5
u_{xCRM-1}	0	0,74	Normal	1,0	0,74
u_{ms}	0	0,29	Rectangular	1,0	0,29
u_{CRM-D}	0	0	Triangular	1,0	0
Combined measurement uncertainty u_{CM}					2,62
Expanded measurement uncertainty U_{CM} (where k = 2)					5,25

B.3 Measurement uncertainty of reference test block

The measurement uncertainty of reference test blocks[3] is given by Formula (B.3):

$$u_{CRM} = \sqrt{u_{CM}^2 + u_{xCRM-2}^2} \quad \text{(B.3)}$$

where

u_{CM}	is the uncertainty of the Leeb hardness calibration machine;
$u_{\times CRM-2}$	is the standard measurement uncertainty due to inhomogeneity of Leeb hardness of reference test block across the surface.

Table B.3 — Determination of inhomogeneity of reference test block

No	**Calculated Leeb hardness value H_{CRM}** HLD
1	764
2	770
3	768
4	768
5	765
6	770
7	766
8	767
9	772
10	771
Mean value	768,1
Standard deviation $s_{\times CRM\text{-}2}$	2,6
Standard measurement uncertainty $u_{\times CRM\text{-}2}$	0,87

Standard uncertainty CRM:

$$u_{\times CRM-2} = \frac{t \cdot s_{\times CRM-2}}{\sqrt{10}} \quad \text{(B.4)}$$

using Student's t-factor t = 1,06 and n = 10:

$u_{\times CRM-2}$ = 0,87 HLD

