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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

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

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

This second edition cancels and replaces the first edition (ISO 4545-2:2005), which has been technically revised.

The main changes compared to the previous edition are as follows:

— all references have been removed of indentation diagonals <0,020 mm;

— the requirements for the calibration and verification of the measuring system have been revised;

— the requirements for the maximum permissible error in measuring a reference indentation have been revised;

— the recommendations for inspection and monitoring of the indenter have been moved to ISO 4545-1;

— Annex A has been revised.

A list of all parts in the ISO 4545 series can be found on the ISO website.

Metallic materials — Knoop hardness test —

Part 2:
Verification and calibration of testing machines

1 Scope

This document specifies the method of verification and calibration of testing machines for determining Knoop hardness for metallic materials in accordance with ISO 4545-1.

A direct method of verification and calibration is specified for the testing machine, indenter, and the diagonal length measuring system. An indirect verification method using reference blocks is specified for the overall checking of the machine.

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

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 376:2011, *Metallic materials — Calibration of force-proving instruments used for the verification of uniaxial testing machines*

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

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

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at https://www.iso.org/obp

4 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 plunger holding the indenter is capable of moving freely without any friction or excessive side play,

b) the indenter is firmly mounted in the plunger,

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

d) the diagonal measuring system

— if integral with the machine, the change in mode from the application and removal of the test force to the diagonal measuring mode does not influence the readings,

— the illumination device of the measuring microscope produces uniform lighting of the whole observed field with enough contrast between the indentation and the surrounding surface to determine the boundary clearly, and

— the centre of the indentation is near the centre of the field of view, if necessary.

NOTE The criteria specified in this document for the performance of the testing machine have been developed and refined over a significant period of time. When determining a specific tolerance that the machine needs to meet, the uncertainty associated with the use of measuring equipment and/or reference standards has been incorporated within this tolerance, and it would therefore be inappropriate to make any further allowance for this uncertainty by, for example, reducing the tolerance by the measurement uncertainty. This applies to all measurements made when performing a direct or indirect verification of the machine.

5 Direct verification

5.1 General

5.1.1 Direct verification shall be conducted in accordance with the schedule given in Clause 7.

5.1.2 Direct verification involves

a) the calibration of the test force,

b) the verification of the indenter,

c) the calibration and verification of the diagonal measuring system, and

d) the verification of the testing cycle.

5.1.3 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, this shall be stated in the verification report.

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

5.2 Calibration of the test force

5.2.1 Each test force to be used within the force range of the testing machine shall be measured. Whenever the indenter position affects the applied force, this shall be done at not less than three positions of the plunger uniformly spaced throughout its range of movement during testing.

For testing machines whose test force is shown not to be influenced by the position of the plunger, e.g. closed-loop controlled loading system, the test force can be calibrated in one position.

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

— by means of an elastic proving device in accordance with ISO 376:2011, class 1, or better;

— by balancing against a force, accurate to ±0,2 %, applied by means of calibrated masses or another method with the same accuracy.

Evidence should be available to demonstrate that the output of the force-proving device does not vary by more than 0,2 % in the period of 1 s to 30 s following a stepped change in force.

5.2.3 Three readings shall be taken for each test force, F, at each position of the plunger. 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 maximum permissible percent relative error, ΔF_{rel}, defined in Table 1.

The percent relative error, ΔF_{rel}, of each measurement of the force, F, is calculated according to Formula (1):

$$\Delta F_{rel} = 100 \times \frac{F - F_{RS}}{F_{RS}} \tag{1}$$

where

F is the measured test force;

F_{RS} is the nominal test force.

Table 1 — Test force tolerances

Range of the nominal test force, F_{RS} N	Maximum permissible relative error, ΔF_{rel} $\%F$
$0{,}009\,807 \le F_{RS} < 0{,}098\,07$	±2,0
$0{,}098\,07 \le F_{RS} < 1{,}961$	±1,5
$1{,}961 \le F_{RS} \le 19{,}613$	±1,0

5.3 Verification of the indenter

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

5.3.2 The verification of the shape of the indenter can be made by direct measurement or optical measurement. The device used for the verification shall have a maximum expanded uncertainty of 0,07°.

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

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

5.3.5 The indenter constant c (see ISO 4545-1:2018, Table 1) shall be within 1,0 % of the ideal value 0,070 28, ($0{,}069\,58 \le c \le 0{,}070\,98$).

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

5.3.6 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°.

5.3.7 The four faces should ideally meet at a common point; however, there is usually a line of junction between opposite faces as shown in Figure 2. The length of the line of junction shall be determined by directly measuring the indenter tip, or by measuring the tip impression in an indentation. The maximum permissible length of the line of junction between opposite faces shall be less than 0,001 mm.

5.3.8 A valid calibration certificate shall exist which confirms the geometrical deviations of the indenter (see 8.2).

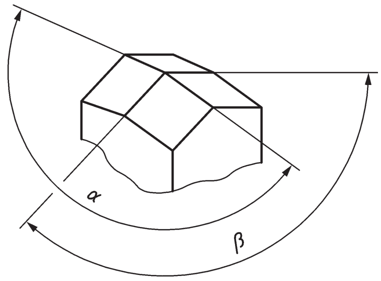

Figure 1 — Indenter geometry

Dimensions in micrometres

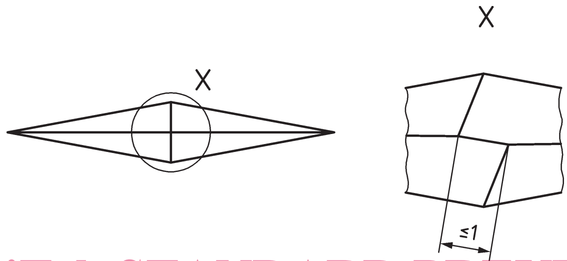

Figure 2 — Line of junction on the top of the indenter (schematic)

5.4 Calibration and verification of the diagonal measuring system

5.4.1 The system for measuring the diagonal of the indentation shall be verified at each magnification and for each incorporated line scale to be used. When an individual scale is used in two perpendicular axes, it shall be calibrated in both orientations. Measurements shall be performed using a calibrated stage micrometer. The maximum expanded uncertainty of the distances between the line intervals on the stage micrometer shall be as indicated in Table 2.

5.4.2 Measurements shall be made at a minimum of four evenly spaced intervals, arranged centrally in the field of view, covering each working range. Three measurements shall be made at each of the evenly spaced intervals. The maximum permissible error of each of the three measurements at each interval shall be as indicated in Table 2.

Table 2 — Calibration and verification requirements of the measuring system

Measurement parameters	Calibration and verification requirements
Maximum expanded uncertainty of the distances between the line intervals on the stage micrometer (see 5.4.1)	Greater of 0,000 4 mm or 0,2 %
Maximum permissible error of the measurements of the stage micrometer intervals (see 5.4.2)	Greater of 0,000 8 mm or 1,0 % of the length measured

5.5 Verification of the testing cycle

The testing cycle shall be timed with equipment having a maximum expanded uncertainty of 1 s. The timing values obtained shall fall within the limits set for the testing cycle in ISO 4545-1.

5.6 Uncertainty of calibration/verification

Uncertainty of the direct verification results shall be determined. An example is given in Annex A.

6 Indirect verification

6.1 General

6.1.1 Indirect verification shall be conducted in accordance with the schedule given in Clause 7.

6.1.2 Indirect verification involves verification of the overall performance of the testing machine by means of reference blocks calibrated in accordance with ISO 4545-3.

6.1.3 Indirect verification should be carried out at a temperature of (23 ± 5) °C. If the verification is made outside this temperature range, this shall be reported in the verification report.

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

6.2 Test force and hardness levels

The testing machine shall be verified by testing reference blocks that have been calibrated in accordance with ISO 4545-3. The blocks shall have been calibrated using the same test forces that the machine will use for future testing. When verifying more than one test force, at least two reference blocks shall be selected from the hardness ranges specified below for each test force that the machine will be verified. The set of blocks needed for verifying the machine for all the test forces shall be chosen so that at least one reference block from each hardness range is used for the verifications. When verifying testing machines using only one test force, three reference blocks shall be used, one from each of the three hardness ranges specified below. The hardness ranges should be chosen, when possible, to replicate the hardness levels most commonly tested when using the specific test forces:

— <250 HK;

— 250 HK to 650 HK;

— >650 HK.

6.3 Measurement of reference indentations

One of the reference indentations from the current calibration period on each reference block shall be measured. For each indentation, the difference between the measured value and the certified diagonal length shall not exceed the greater of 0,001 mm and 1,25 % of the reference indentation length. If preferred, this check may instead be made on a similarly-sized indentation in a different reference block with similar hardness.

6.4 Number of indentations

On each reference block, five indentations shall be made and measured. The tests shall be carried out in accordance with ISO 4545-1. Only the calibrated surfaces of the test blocks are to be used for testing.

6.5 Verification result

For each reference block, let H_1, H_2, H_3, H_4, H_5 be the measured hardness arranged in increasing order of magnitude, corresponding to the measured diagonals d_1, d_2, d_3, d_4, d_5 in decreasing order of magnitude. The mean hardness value, $\overline{H}$, is calculated according to Formula (2), and the mean diagonal length, $\overline{d}$, is calculated according to Formula (3):

$$\overline{H} = \frac{H_1 + H_2 + H_3 + H_4 + H_5}{5} \tag{2}$$

$$\overline{d} = \frac{d_1 + d_2 + d_3 + d_4 + d_5}{5} \tag{3}$$

6.6 Repeatability

The relative repeatability of the testing machine, r_{rel}, expressed as a percentage of $\overline{H}$, is calculated according to Formula (4):

$$r_{rel} = 100 \times \frac{H_5 - H_1}{\overline{H}} \tag{4}$$

The repeatability of the testing machine is satisfactory if $(d_1 - d_5) \leq 0{,}001$ mm. If $(d_1 - d_5) > 0{,}001$ mm, the testing machine is satisfactory if r_{rel} is less than or equal to the percentages indicated in Table 3.

Table 3 — Maximum permissible relative repeatability

Knoop hardness of the reference block	**Maximum permissible relative HK repeatability of the testing machine, r_{rel}** %HK	
	HK 0,5 to HK 2	HK 0,001 to <HK 0,5
$100 \leq$ HK ≤ 250	16,0	18,0
$250 <$ HK ≤ 650	10,0	10,0
HK > 650	8,0	8,0
NOTE Lower hardness materials often exhibit higher values of repeatability than those for higher hardness materials.		

6.7 Bias

The bias, b, of the testing machine under the particular verification conditions is calculated according to Formula (5):

$$b = \overline{H} - H_{CRM} \tag{5}$$

where

H_{CRM} is the certified hardness of the reference block used.

The percent bias, b_{rel}, is calculated according to Formula (6):

$$b_{rel} = 100 \times \frac{\overline{H} - H_{CRM}}{H_{CRM}} \tag{6}$$

The maximum positive or negative bias of the testing machine, expressed as a percentage of the specified hardness of the reference block, shall not exceed the values given in Table 4.

Table 4 — Maximum permissible percent HK bias

Mean diagonal length $\bar{d}$ mm	Maximum permissible percent HK bias, b_{rel}, of the testing machine, ± %HK
$0{,}02 \le \bar{d} < 0{,}06$	$0{,}24/\bar{d}$
$0{,}06 \le \bar{d}$	4

6.8 Uncertainty of calibration/verification

The uncertainty of the calibration results shall be determined. An example is given in Annex A.

7 Intervals between verifications

Direct verifications shall be performed according to the schedule given in Table 5. It is recommended that direct verifications be performed every 12 months.

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

Table 5 — Direct verifications of hardness testing machines

Requirements of verification	Force	Diagonal measuring system	Test cycle	Indenter[a]
Before setting to work first time	x	x	x	x
After dismantling and reassembling, if force, diagonal measuring system or test cycle are affected	x	x	x	—
Failure of indirect verification[b]	x	x	x	—
Indirect verification > 13 months ago	x	x	x	—

[a] In addition, it is recommended that the indenter be directly verified after two 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.

8 Verification report/calibration certificate

8.1 Knoop testing machine

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

a) a reference to this document, i.e. ISO 4545-2;

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

c) an identification data of the hardness testing machine;

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

e) the test force(s) verified;

f) the hardness values of reference blocks used;

g) the verification temperature, if it is outside the range specified in 5.1.3;