INTERNATIONAL STANDARD

ISO 4545-2

Second edition 2017-12

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).

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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.

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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 <u>Clause 7</u>.
- **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 ineasuring 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.

uillib.

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):

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

where

F is the measured test force.

 F_{RS} is the nominal test force

Table 1 — Test force tolerances

***************************************	n normiccible relative
	error, $\Delta F_{\rm rel}$
	%F
Amminimi, in	±2,0
	±1,5
	±1,0
Contract to the contract of th	7

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 $(172.5 \pm 0.1)^{\circ}$ (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 $(130 \pm 1,0)^{\circ}$ (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 $\pm 0.5^{\circ}$.
- **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 <u>Figure 2</u>. 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).

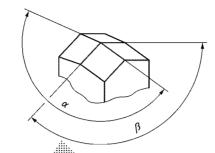
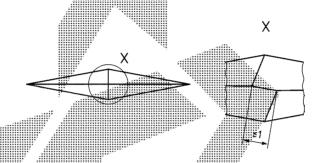


Figure 1 — Indenter geometry



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.

Indirect verification

6.1 General

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

Test force and hardness levels 6.2

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;</p>
- 250 HK to 650 HK;
- >650 HK.

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, H, is calculated according to Formula (2), and the mean diagonal length, \overline{d} , is calculated according to Formula (3):

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

$$\frac{-d_1 + d_2 + d_3 + d_4 + d_5}{5} \tag{3}$$

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):

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

The repeatability of the testing machine is satisfactory if $(d_1 - d_5) \le 0.001$ mm. If $(d_1 - d_5) > 0.001$ mm, the testing machine is satisfactory if $r_{\rm rel}$ is less than or equal to the percentages indicated in Table 3.

Table 3 — Maximum permissible relative repeatability

***************************************	*******	, , , , , , , , , , , , , , , , , , , ,
Knoop hardness of th	Maximum permissible r	elative HK repeatability machine, r _{rel}
reference block		
1		HK 0,001 to <hk 0<="" b="">,5</hk>
100 ≤ 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):

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

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 <u>Table 4</u>.

Table 4 — Maximum permissible percent HK bias

Mean diagonal length d mm	Maximum permissible percent HK bias, $b_{ m rel}$, of the testing machine, $\pm\%{ m HK}$			
$0.02 \le \frac{-}{d} < 0.06$ iii.	$0,24/\overline{d}$			
0,06 ≤ 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 <u>Table 5</u>. 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

444444444444444444444444444444444444444	***************		,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,	27.77.77.77.77.77.77.77.77.77.77.77.77.7	
Requirements of verificati	on	Force	Diagonal measuring system	Test cycle	Indentera
Before setting to work first time		×	x	X.	X
After dismantling and reassembling if force, diagonal measuring system test cycle are affected	giiiiiiiiiiiiiiiiiiiiiiiiiiiiiiiiiiiii	X	X	X	***************************************
Failure of indirect verification ^b	***************************************	X	х	X	_
Indirect verification > 13 months a	go	X	X	х	

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

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;

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.

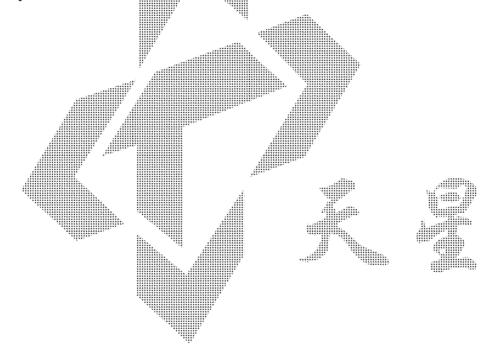
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- h) all results obtained;
- i) the date of verification and reference to the verification institution;
- j) the uncertainty of the verification result.

8.2 Knoop indenter

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

- a) a reference to this document, i.e. ISO 4545-2;
- b) an indenter serial number;
- c) all results obtained;
- d) the date of verification and reference to the verification institution;
- e) the uncertainty of the verification **result**



Annex A

(informative)

Uncertainty of the calibration results of the hardness testing system

A.1 General

Measurement uncertainty analysis is a useful tool to help determine sources of error and to understand differences between measured values. This annex gives guidance on uncertainty estimation but the methods contained are for information only, unless specifically instructed otherwise by the customer. 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. In each case, it is simply the measured value resulting from the use of the specified measuring equipment and/or reference standards that is used to assess whether or not the machine complies with this document. However, there may be special circumstances where reducing the tolerance by the measurement uncertainty is appropriate. This should only be done by agreement of the parties involved.

The metrological chain necessary to define and disseminate hardness scales is discussed in ISO 4545-1.

A.2 Direct verification of the hardness testing machine

A.2.1 Calibration of the test force

The combined relative standard uncertainty of the test force calibration is calculated according to Formula (A.1):

$$u_{\rm F} = \sqrt{u_{\rm FRS}^2 + u_{\rm FHTM}^2} \tag{A.1}$$

where

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

 $u_{\text{FHTM},i}$ 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

Using the results as shown in <u>Table A.1</u> and the following information:

- uncertainty of measurement of the force transducer (from calibration certificate): $U_{FRS} = 0.24 \%$ (k = 2), and
- calibration value of the force transducer F_{RS} = 9,806 7 N,

the combined relative standard uncertainty of the test force calibration, u_F , is calculated according to Formula (A.1) and as shown in Table A.2.

In Table A.1, the relative deviation is calculated according to Formula (A.2):

$$\Delta F_{\text{rel},i,j} = \frac{F_{i,j} - F_{RS}}{F_{RS}} \tag{A.2}$$

where

 $F_{i,j}$ is the test force measurement value in the *i*-th height position of the *j*-th measurement series.

The relative standard uncertainty is calculated according to Formula (A:3)

$$u_{\text{FHTM},i} = \frac{s_{F_i}}{F_i} \times \frac{t_i}{\sqrt{n}}, (n = 3, t = 1,32) \tag{A.3}$$

where s_{F_i} is the standard deviation of the test-force indication values in the *i*-th height position.

Table A.1 — Results of the test force calibration

			Series 2		Series 3				
Number of height position for test force calibration	Value F _{i,1}		ratue F ₁₂		Value <i>F</i> _{<i>i</i>,3}	Relative deviation $\Delta F_{\mathrm{rel},i,3}$	Mean walue	Standard deviation s_{F_i}	Relative standard uncertainty u _{FHTM,i}
	N	%	N "	%	N	%	N		
1	9,809	0,023	9,815	0,085	9,822	0,156	9,815	0,006 51	0,51 × 10− ³
2	9,802	-0,048	9,817	0,105	9,820	0,136	9,813	0,009 64	0,75 × 10 ^{−3}
3	9,810	0,034	9,812	0,054	9,828	0,217	9,817	0,009 87	0,77 × 10−3

In <u>Table A.2</u>, the uncertainty of the measurement of the test force is determined using the maximum value of relative standard uncertainty, $u_{\text{FHTM},i}$, from <u>Table A.1</u> (in this case, $u_{\text{FHTM},3}$).

Quantity X_p	Estimated value x_p	Relative limit values a_p	Distribution type	Symbol	Relative standard uncertainty $u(x_p)$	Sensitivity coefficient c_p	Relative uncertainty contribution $u_p(F)$	
Force transducer indication	9,806 7 N	±0,24 %	Normal	u_{FRS}	1,2 × 10−3	1	1,2 × 10−3 N	
Accuracy of generated force	0 N	±1 %:	Normal	иғнтм	0,77 × 10 ⁻³	1	0,77 × 10 ⁻³ N	
Relative combined standard uncertainty $u_{ m F}$, %								
Relative expanded uncertainty of measurement $U_{\mathbb{R}}: \mathbb{R} = 21.26$								

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

In Table A.3, the maximum relative deviation of the test force including the uncertainty of measurement of the reference instrument is calculated according to Formula [A.4] using the maximum absolute value of $\Delta F_{\text{rel},i,j}$ from Table A.1 (in this case, $\Delta F_{\text{rel},3,3}$).

$$\Delta F_{\text{max}} = |\Delta F_{\text{rel},i,j,j}| + U_{\text{F},i,j} \tag{A.4}$$

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

Maximum relative deviation of test force $\Delta F_{\mathrm{rel},3,3}$	measurement unc		Max. relative deviation of test force including measurement uncertainty of reference instrument $\Delta F_{\rm max}$
0,217 %	0,285	%	0,502 %

where ΔF_{max} is calculated according to Formula (A.5):

$$\Delta F_{\text{max}} = |\Delta F_{\text{rel}}| + U_{\text{F}} \tag{A.5}$$

The result of the example means that the deviation of the test forces including the uncertainty of measurement of the reference instrument specified in 5.2, amounting to ± 1.0 % is complied with.

A.2.2 Calibration of the diagonal measuring system

The combined relative standard uncertainty of the reference instrument for the diagonal measuring system is calculated according to <u>Formula (A.6)</u>:

$$u_{\rm L} = \sqrt{u_{\rm LRS}^2 + 2 \times u_{\rm ms}^2 + u_{\rm LHTM}^2} \tag{A.6}$$

where

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

 $u_{\rm ms}$ is the uncertainty of measurement due to the resolution of the diagonal measuring system;

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

Regarding $u_{\rm ms}$, both the resolution of the length measurement indicating instrument and the optical resolution of the measuring microscope shall be considered. In most cases, the overall resolution of the

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measurement system should be included twice in the calculation of u_L due to resolving the positions of both the zero and indicator lines of the stage micrometer.

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

- temperature dependence,
- long-term stability, and
- interpolation deviation.

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

EXAMPLE

For this example, four indication intervals are measured (0,05 mm, 0,10 mm, 0,15 mm, and 0,20 mm).

Using results as shown in Table A.4 and the following information:

uncertainty of measurement of the stage micrometer.

 $U_{LRS} = 0.000 \text{ 1 mm } (k = 2)$

- optical resolution of the microscope objective:

 $\delta_{\rm OR}$ = 0,000 34 mm

NOTE $\delta_{0R} = \lambda / (2 \times NA)$, where

- λ = the wave length of light in μ m (approx. 0,55 μ m for green light), and
- MA = the numerical aperture of the objective.

EXAMPLE: For a 100× objective with an NA of 0,8 using green light, δ_{OR} = 0,55 μ m/ (2 × 0,8) = 0,34 μ m.

The above optical resolution for mula is valid for lighting inclined at large angles.

- resolution of the display integrator of the measuring system: $\delta_{IR} = 0,000 \text{ 1 mm}$;
- the resolution of the diagonal measuring system is calculated according to Formula (A.7):

$$\delta_{\rm ms} = \sqrt{\delta_{\rm OR}^2 + \delta_{\rm IR}^2} = 0,00035 \,\rm mm$$
 (A.7)

the combined relative standard uncertainty of the reference instrument for the diagonal measuring system, u_L , is calculated according to <u>Formula (A.6)</u>, and as shown in <u>Table A.5</u>.

In <u>Table A.4</u>, the relative deviation $\Delta L_{\text{rel},i,j}$ for the *i*-th indication interval of the *j*-th measurement series of the stage micrometer is calculated according to <u>Formula (A.8)</u>:

$$\Delta L_{\text{rel},i,j} = \frac{L_{i,j} - L_{\text{RS},i}}{L_{\text{RS},i}} \times 100 \tag{A.8}$$

where

 $L_{RS,i}$ is the *i*-th indication interval value of the stage micrometer;

 $L_{i,j}$ is the length measurement value for the *i*-th indication interval of the *j*-th measurement series of the stage micrometer.

		Series 1		Series 2		Series 3			
Number of indication interval	Indication interval value (i) of the stage micrometer $L_{RS,i}$	Value L _{i,1}	Relative deviation $\Delta L_{{ m rel},i,1}$	Value L _{i,2}	Relative deviation $\Delta L_{{ m rel},i,2}$	Value $L_{i,3}$	Relative deviation $\Delta L_{{ m rel},i,3}$	Mean value \overline{L}_i	Standard deviation SLi
	mm	mm _	ii %	mm	%	mm	%	mm	mm
1	0,05	0,050 1	0,200	0,050 0	0,000	0,049 9	-0,200	0,0500	0,000 100
2	0,10	0,1002	0,200	0,100 0	0,000	0,100 1	0,100	0,100 1	0,000 100
3	0,15	0,1501	0,067	0,149 6	-0,267	0,150 1	0,067	0,149 9	0,000 289
4	0,20	0,1997	-0,150	0,200 1	0,050	0,200 1	0,050	0,2000	0,000 231

Table A.4 — Results of the calibration of the diagonal measuring system

The standard uncertainty of measurement of the diagonal measuring system u_L is calculated in accordance with Formula (A.6). The contributions to the calculation are given in <u>Table A.5</u>, and calculated as follows.

The relative standard uncertainty of measurement of the stage micrometer for the *i*-th indication interval is calculated according to Formula (A.9):

$$u_{\text{LRS},i} = \frac{U_{\text{LRS},i}}{U_{\text{RS},i}} \tag{A.9}$$

The relative standard uncertainty due to the resolution of the diagonal measuring system for the *i*-th indication interval is calculated according to Formula (A.10):

$$u_{\text{ms},i} = \frac{\delta_{\text{ms}}^{\text{ms}}}{L_{\text{RS},i}} \tag{A.10}$$

The relative standard uncertainty of the measurements of the i-th indication interval is calculated according to Formula (A.11):

$$u_{\text{LHTM},i} = \frac{s_{Li}}{L_i} \times \frac{t}{\sqrt{n}} \ (n = 3, t = 1,32)$$
 (A.11)

where

 \mathbf{s}_{Li} is the standard deviation of the length measurement values for the i-th indication interval of the stage micrometer;

 \overline{L}_{c} is the mean length measurement value for the i-th indication interval of the stage micrometer.

Table A.5 — Calculation of the uncertainty of measurement of the diagonal measuring system

Quantity X_p	Distribution type	Estimated value	Symbol	Formula: relative standard uncertainty $u(x_p)$	Sens. coeff. c_p	Relative uncertainty contribution, $u_p(L)$			n, u _p (L)
						L _{RS,1} (0,05 mm)	L _{RS,2} (0,10 mm)	L _{RS,3} (0,15 mm)	L _{RS,4} (0,20 mm)
Stage micrometer	Normal	$L_{\mathrm{RS},i}$	$u_{ m LRS}$	$u_{\text{LRS},i} = \frac{U_{\text{LRS}}}{2}$ $L_{\text{RS},i}$	1	1,00 × 10 ⁻³	0,50 × 1 010 ⁻³	0,33 × 10 ⁻³	0,25 × 10 ^{−3}
Resolution of the diagonal measuring system (include twice)	Rectangular	0,000 35 mm	u _{ms} .:	$u_{\text{med}} = \frac{3_{\text{max}}}{L_{\text{RS},i}}$	1	2,28 × 10 ⁻³	1,14 × 10−3	0,76 × 10−3	0,57 × 10 ^{−3}
Accuracy of the diag- onal measuring system	Normal	$L_{\mathrm{RS},i}$	$u_{ m LHTM}$	$u_{\text{LHTM}} = \frac{s_{\text{LHTM}}}{m} \times \frac{t}{\sqrt{n}}$ $\left(n = 3.t = 1.32\right)$	1	1,52×10- ³	0,76 × 10−3	1,47 × 10-3	0,88 × 10 ⁻³
Relative com	bined uncertain	ty af measure	me nt u z,	и		0,370	0,185	0,185	0,122
Relative expa	anded uncertain	ty of measure	ement, U _{L,i}	(k = Z), %		0,740	0,370	0,370	0,244

In <u>Table A.6</u>, the <u>maximum</u> relative deviation of the diagonal measuring system including the uncertainty of measurement of the length reference instrument, $\Delta L_{\max,i}$, for the *i*-th-indication interval of the stage micrometer is calculated according to Formula (A.12):

$$\Delta L_{\max,i} = \max \left| \Delta L_{\text{rel},i} \right| + U_{\text{L},i} \tag{A.12}$$

where $\max |\Delta L_{\mathrm{rel},i}|$ is the maximum absolute value of the relative deviations $\Delta L_{\mathrm{rel},i,j}$ for the i-th indication interval of the stage micrometer from Table A.4.

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

Number of indication interval	Indication interval value (i) of the stage micrometer $L_{{ m RS},i}$	Maximum relative deviation of the diagonal measuring system $\max \Delta L_{\mathrm{rel},i} $	Expanded relative uncertainty of measurement $U_{\mathcal{L}_i}$	Max. relative deviation of diagonal measuring system including measurement uncertainty of length reference instrument $\Delta L_{ m max,i}$	Maximum permissible relative deviation specified in
1	0,05 mm	0,20 %	0,74 %	0,94 %	1,2 %
2	0,10 mm	0,20 %	0,37 %	0,57 %	1,0 %
3	0,15 mm	0,27 %	0,37 %	0,64 %	1,0 %
4	0,20 mm	0,15 %	0,24 %	0,39 %	1,0 %

The result of the example means that the deviation of the diagonal measuring system, including the uncertainty of measurement of the length reference instrument specified in 5.4 is complied with for each of the indication intervals of the stage micrometer.

A.2.3 Verification of the indenter

The indenter, consisting of indenter tip and holder, cannot be verified or respectively calibrated in-site. (see 5.3).

A.2.4 Verification of the test cycle

While measuring with a usual time measuring system (stopwatch), the uncertainty can be indicated as 0,1 s. Therefore, an estimation of the uncertainty of measurement is not necessary.

A.3 Indirect verification of the hardness testing machine

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 results of measuring the reference indentation may be used to evaluate the uncertainty of the diagonal measuring system.

NOTE In this annex, the index "CRM (certified reference material)" means, according to the definitions of the hardness testing standards, "hardness reference block".

The uncertainty of measurement of the indirect verification of the hardness testing machine is calculated according to Formula (A.13):

$$u_{\rm HTM} = \sqrt{u_{\rm CRM}^2 + u_{\rm CRM-D}^2 + u_{\rm HCRM}^2 + 2 \times u_{\rm ms}^2}$$
 (A.13)

where

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

*u*_{CRM-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);

 $u_{\rm HCRM}$ is the standard uncertainty of the hardness testing machine when measuring the CRM;

 $u_{
m ms}$ is the uncertainty due to the resolution of the diagonal measuring system of the hardness testing machine. Both the resolution of the length measurement indicating instrument and the optical resolution of the measuring microscope shall be considered. In most cases, the overall resolution of the measurement system should be included twice in the calculation of $u_{
m HTM}$ due to resolving the positions of both ends of the long diagonal independently.

EXAMPLE

Using results as shown in <u>Table A.7</u> and the following information:

— hardness of the hardness reference block $H_{CRM} = 802,7 \text{ HK } 1$

— uncertainty of measurement of the hardness reference block $U_{CRM} = \pm 12,0 \text{ HK } 1$

- resolution of the diagonal measuring system of the hardness testing machine,

 $\delta_{\rm ms}$ = 0,000 35 mm

calculated according to Formula (A.14),

$$\delta_{\rm ms} = \sqrt{\delta_{\rm OR}^2 + \delta_{\rm IR}^2} \tag{A.14}$$

where

 δ_{OR} is the optical resolution of the microscope objective (0,000 34 mm) (see the example in A.2.2);

 δ_{IR} is the resolution of the display indicator of the measuring system (0,000 1 mm);

 u_{ms} is the standard uncertainty of measurement due to the resolution of the diagonal measuring system (rectangular distribution) $\left(\frac{0,000\,35\,\mathrm{mm}}{2\sqrt{3}} = 0,000\,102\,\mathrm{mm}\right)$,

the uncertainty of measurement of the indirect verification of the hardness testing machine, u_{HTM} , is calculated according to Formula (A.13) and as shown in Table A.8.

Measured indentation Calculated hardness value. diagonal, Number. Н d HK1 mm . 0,133 2 802.0 0,1333 8.008 0.1335_{max}798,4_{min} 804,4_{max} $0,1330_{\min}$ 803.2 0,1331 801,7 0,1332 Mean value, H 2,3 Standard deviation, s_H HK: Knoop hardness

Table A.7 — Results of the indirect verification

The bias is calculated according to Formula (A.15):

$$b = \overline{H} - H_{CRM} \tag{A.15}$$

$$b = 801,7 - 802,7 = -1,0 \text{ HK } 1$$

The standard uncertainty of the hardness testing machine when measuring the CRM is calculated according to $\underline{Formula\ (A.16)}$:

$$u_{\text{HCRM}} = \frac{t \times s_{\text{H}}}{\sqrt{n}} \tag{A.16}$$

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

 $u_{\rm HCRM} = 1.18~{\rm HK}~1$

A.4 Budget of uncertainty of measurement

The contribution of each element to the combined uncertainty of measurement is given in <u>Table A.8</u> and the maximum deviation of the hardness testing machine including the uncertainty of measurement is shown in <u>Table A.9</u>.

 $Table \ A.8 -- Budget \ of \ uncertainty \ of \ measurement$

$\begin{array}{c} \textbf{Quantity} \\ X_p \end{array}$	Estimated value x_p	Standard uncertainty of measurement $u(x_p)$	Distribution type	Sensitivity coefficient c_p	Uncertainty contribution $u_p(H)$ HK 1		
$u_{\rm CRM}$	802,7 HK 1	6,0 HK 1	Normal	1,0	6,0		
$u_{ m HCRM}$	802,7 HK 1	1,18 HK 1	Normal	1,0	1,18		
$u_{ m ms}$	0 mm	0,000 102 mm	Rectangular	-12 057,9a	-1,23		
u _{CMR-D}	0 HK 1	0 HK 1	Triangular	1,0	0		
Combined uncert	6,36						
Expanded uncert	12,7						
HK: Knoop hardness The sensitivity coefficient follows from $c = \partial H/\partial d = -2(H/d)$ for $H = 801,7$ HK 1 and $d = 0,133$ 2 mm.							

The maximum deviation of the testing machine including the uncertainty of measurement is calculated

according to Formula (A.17) as shown in Table A.9

$$\Delta H_{\text{HTMmax}} = \ddot{U}_{\text{HTM}} + \dot{b} = 12,7 \text{ HK} 1 + 1,0 \text{ HK} 1 = 13,7 \text{ HK} 1$$
 (A.17)

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

				,
Measured hardness tes the hardness tes machine H	ess on sting	Expanded uncertainty of measurement U _{HTM}	calibrating with the reference block	Maximum deviation of the testing machine including uncertainty of measurement $\Delta H_{\mathrm{HTMmax}}$
801,7 HK 1	***************************************	iiiiiiiiiiiiiiiiiiiiiiiiiiiiiiiiiiiiii	1,0 HK 1	13,7 HK 1
HK: Knoop hardness	***************************************			

The relative deviation of the testing machine including uncertainty of measurement is calculated according to Formula (A.18):

$$\frac{\Delta H_{\text{HTMmax}}}{\overline{H}} = \frac{13.7}{801.7} = 1.7 \% \tag{A.18}$$

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