INTERNATIONAL STANDARD

# ISO 4545-1

Second edition
2017-12

# Metallic materials — Knoop hardness test —

## Part 1: Test method

*Matériaux métalliques — Essai de dureté Knoop —*

*Partie 1: Méthode d'essai*

Reference number
ISO 4545-1:2017(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.

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 4545-1:2005), 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 resolution requirements have been defined for the measuring system;

— the lower test force limit of the Knoop hardness test has been expanded to 0,009 807 N;

— the requirements for the periodic (weekly or daily) verifications of the testing machine have been defined as normative, the maximum permissible bias value has been revised, and 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 added (moved from ISO 4545-2);

— the requirements have been revised for the approach velocity of the indenter prior to contact with the sample surface;

— the timing requirements for the test force application and the duration at maximum test force are revised to indicate target time values;

— Figure 3 has been added illustrating the requirements for the minimum distance between indentations; the distances have been stated with respect to the indentation centres rather than the indentation limits, but the requirements have not changed;

— the requirements have been added to the test report for reporting the test date and any hardness conversion method used;

 

— Annexes C, D and E have been added concerning Knoop hardness measurement traceability, the CCM — Working group on hardness and adjustment of Köhler illumination systems, respectively.

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



 

# Metallic materials — Knoop hardness test —

# Part 1:
# Test method

## 1 Scope

This document specifies the Knoop hardness test method for metallic materials for test forces from 0,009 807 N to 19,613 N.

The Knoop hardness test is specified in this document for lengths of indentation diagonals ≥0,020 mm. Using this method to determine Knoop hardness from smaller indentations is outside the scope of this document as results would suffer from large uncertainties due to the limitations of optical measurement and imperfections in tip geometry. ISO 14577-1 allows the determination of hardness from smaller indentations.

A periodic verification method is specified for routine checking of the testing machine in service by the user.

Special considerations for Knoop testing of metallic coatings can be found in ISO 4516.

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

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 http://www.iso.org/obp

## 4 Principle

A diamond indenter, in the form of a rhombic-based pyramid with angles, $\alpha$ and $\beta$, between opposite edges respectively equal to 172,5° and 130° at the vertex, is forced into the surface of a test piece followed by measurement of the long diagonal, $d$, of the indentation remaining in the surface after removal of the test force, $F$ (see Figures 1 and 2).

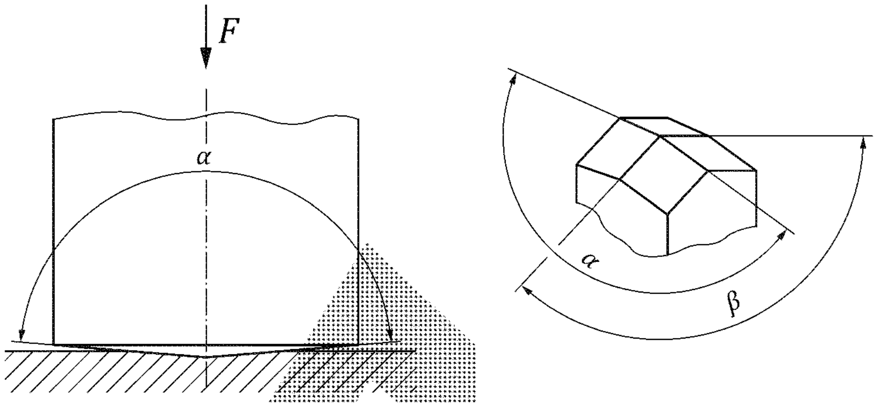


**Figure 1 — Principle of the test and indenter geometry**

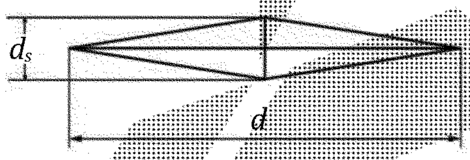


**Figure 2 — Knoop indentation**

The Knoop hardness is proportional to the quotient obtained by dividing the test force by the projected area of the indentation, which is assumed to be a rhombic-based pyramid, and having at the vertex the same angles as the indenter.

NOTE As applicable, this test document has adopted hardness test parameters as defined by the working group on hardness (CCM-WGH) under the framework of the International Committee of Weights and Measures (CIPM) Consultative Committee for Mass and Related Quantities (CCM) (see Annex D).

# 5 Symbols and designations

## 5.1 Symbols and designations used in this document

See Table 1 and Figures 1 and 2.

**Table 1 — Symbols and designations**

| Symbol | Designation |
|---|---|
| $F$ | Test force, in newtons (N) |
| $d$ | Length of the long diagonal, in millimetres |
| $d_s$ | Length of the short diagonal, in millimetres |
| $\alpha$ | Angle between the opposite edges of the long diagonal at the vertex of the diamond pyramid indenter (nominally 172,5°) (see Figure 1) |
| $\beta$ | Angle between the opposite edges of the short diagonal at the vertex of the diamond pyramid (nominally 130°) (see Figure 1) |
| NOTE Standard acceleration due to gravity, $g_n$ = 9,806 65 m/s$^2$, which is the conversion factor from kgf to N.<br>To reduce uncertainty, the Knoop hardness can be calculated using the actual indenter angles $\alpha$ and $\beta$. | |

**Table 1** *(continued)*

| Symbol | Designation |
|---|---|
| $V$ | Magnification of the measuring system |
| $c$ | Indenter constant, relating projected area of the indentation to the square of the length of the long diagonal<br>Indenter constant, $c = \dfrac{\tan\frac{\beta}{2}}{2\tan\frac{\alpha}{2}}$, for nominal angles $\alpha$ and $\beta$, $c$ is approximately 0,070 28 |
| HK | Knoop hardness $= \dfrac{\text{Test force (kgf)}}{\text{Projected area of indentation (mm}^2\text{)}}$<br>$= \dfrac{1}{g_n} \times \dfrac{\text{Test force (N)}}{\text{Projected area of indentation (mm}^2\text{)}}$<br>$= \dfrac{1}{g_n} \times \dfrac{F}{cd^2}$<br>For the nominal indenter constant $c \approx 0{,}070\ 28$,<br>Knoop hardness $= 1{,}451 \times \dfrac{F}{d^2}$ |
| NOTE Standard acceleration due to gravity, $g_n$ = 9,806 65 m/s$^2$, which is the conversion factor from kgf to N.<br>To reduce uncertainty, the Knoop hardness can be calculated using the actual indenter angles $\alpha$ and $\beta$. | |

## 5.2 Designation of hardness number

Knoop hardness, HK, is designated as shown in the following example.

640 HK 0,1 /20

- /20 — Duration of test force (20 s) if not within the specified range (10 s to 15 s)
- 0,1 — Approximate kgf equivalent value of applied test force where (0,1 kgf = 0,980 7 N)
- HK — Hardness symbol
- 640 — Knoop hardness value

# 6 Testing machine

## 6.1 Testing machine

The testing machine shall be capable of applying a predetermined force or forces within the desired range of test forces, in accordance with ISO 4545-2.

## 6.2 Indenter

The indenter shall be a diamond in the shape of a rhombic-based pyramid, as specified in ISO 4545-2.

## 6.3 Diagonal measuring system

The diagonal measuring system shall satisfy the requirements in ISO 4545-2.

Magnifications should be provided so that the diagonal can be enlarged to greater than 25 % but less than 75 % of the maximum possible optical field of view. Many objective lenses are non-linear towards the edge of the field of view.

NOTE A diagonal measuring system using a camera for measurement can use 100 % of the camera's field of view, provided it is designed to consider field of view limitations of the optical system.

The resolution required of the diagonal measuring system depends on the size of the smallest indentation to be measured, and shall be in accordance with Table 2. In determining the resolution of the measuring system, the resolution of the microscope optics, the digital resolution of the measuring scale and the step-size of any stage movement, where applicable, should be taken into account.

**Table 2 — Resolution of the measuring system**

| Diagonal length<br>$d$<br>mm | Resolution of the measuring system |
|---|---|
| $0{,}020 \leq d < 0{,}080$ | 0,000 4 mm |
| $0{,}080 \leq d$ | 0,5 % of $d$ |

# 7 Test piece

## 7.1 Test Surface

The test shall be carried out on a polished surface, which is smooth and even, free from oxide scale and foreign matter and, in particular, free from lubricants, unless otherwise specified in product standards. The finish of the surface shall permit accurate determination of the diagonal length of the indentation.

## 7.2 Preparation

Surface preparation shall be carried out in such a way as to prevent surface damage, or alteration of the surface hardness due to excessive heating or cold-working.

Due to the small depth of Knoop hardness indentations, it is essential that special precautions be taken during preparation. It is recommended to use a polishing/electropolishing technique that is adapted to the material to be measured.

## 7.3 Thickness

The thickness of the test piece, or of the layer under test, shall be at least 1/3 times the length of the diagonal length of the indentation. No deformation shall be visible at the back of the test piece after the test.

NOTE The depth of the indentation is approximately 1/30 of the diagonal length (0,033 $d$).

## 7.4 Support of unstable test pieces

For a test piece of small cross-section or of irregular shape, either a dedicated support should be used or it should be mounted in a similar manner to a metallographic micro-section in appropriate material so that it is adequately supported and does not move during the force application.

# 8 Procedure

## 8.1 Test temperature

The test is normally carried out at ambient temperature within the limits of 10 °C to 35 °C. If the test is carried out at a temperature outside this range, it shall be noted in the test report. Tests carried out under controlled conditions shall be made at a temperature of (23 ± 5) °C.

## 8.2 Test force

The test forces given in Table 3 are typical. Other test forces may be used. Test forces shall be chosen that result in indentations with a long diagonal greater than 0,020 mm.

**Table 3 — Typical test forces**

| Hardness scale | Test force value, *F* | |
|---|---|---|
| | N | Approximate kgf[a] equivalent |
| HK 0,001 | 0,009 807 | 0,001 |
| HK 0,002 | 0,019 61 | 0,002 |
| HK 0,005 | 0,049 03 | 0,005 |
| HK 0,01 | 0,098 07 | 0,010 |
| HK 0,02 | 0,196 1 | 0,020 |
| HK 0,025 | 0,245 2 | 0,025 |
| HK 0,05 | 0,490 3 | 0,050 |
| HK 0,1 | 0,980 7 | 0,100 |
| HK 0,2 | 1,961 | 0,200 |
| HK 0,3 | 2,942 | 0,300 |
| HK 0,5 | 4,903 | 0,500 |
| HK 1 | 9,807 | 1,000 |
| HK 2 | 19,613 | 2,000 |

[a] Not an SI unit.

## 8.3 Periodic verification

The periodic verification defined in Annex A shall be performed within a week prior to use for each test force used but is recommended on the day of use. The periodic verification is recommended whenever the test force is changed. The periodic verification shall be done whenever the indenter is changed.

## 8.4 Test piece support

The test piece shall be placed on a rigid support. The support surfaces shall be clean and free from foreign matter (scales, oil, dirt, etc.). It is important that the test piece lies firmly on the support so that any displacement that affects the test result cannot occur during the test.

## 8.5 Focus on test surface

The diagonal measuring system microscope shall be focused so that the specimen surface and the desired test location can be observed.

NOTE Some testing machines do not require that the microscope be focused on the specimen surface.

## 8.6 Test force application

The indenter shall be brought into contact with the test surface and the test force shall be applied in a direction perpendicular to the surface, without shock, vibration or overload, until the applied force attains the specified value. The time from the initial application of the force until the full test force is reached shall be $7^{+1}_{-5}$ s.

NOTE 1 The requirements for the time durations are given with asymmetric limits. For example, $7^{+1}_{-5}$ s indicates that 7 s is the nominal time duration, with an acceptable range of not less than 2 s (calculated as 7 s – 5 s) to not more than 8 s (calculated as 7 s + 1 s).

The indenter shall contact the test piece at a velocity of ≤0,070 mm/s.

The duration of the test force shall be $14^{+1}_{-4}$ s, except for tests on materials whose time-dependent properties would make this an unsuitable range. For these tests, this duration shall be specified as part of the hardness designation (see 5.2).

NOTE 2 There is evidence that some materials are sensitive to the rate of straining which causes changes in the value of the yield strength. The corresponding effect on the termination of the formation of an indentation can make alterations in the hardness value.

## 8.7 Prevention of the effect of shock or vibration

Throughout the test, the testing machine shall be protected from shock or vibration[6].

## 8.8 Minimum distance between adjacent indentations

The minimum distance between adjacent indentations and the minimum distance between an indentation and the edge of the test piece are shown in Figure 3.

The minimum distance between the edge of the test piece and the centre of any indentation oriented parallel to the edge of the test piece shall be at least 3,5 times the length of the short diagonal of the indentation. The minimum distance between the edge of the test piece and the centre of any indentation oriented perpendicular to the edge of the test piece shall be at least equal to the length of the long diagonal of the indentation.

The minimum distance between the centres of two adjacent indentations, oriented side-by-side, shall be at least 3,5 times the length of the short diagonal. For indentations oriented end-to-end, the minimum distance between the centres of two adjacent indents shall be at least twice the length of the long diagonal. If two indentations differ in size, the minimum spacing shall be based on the diagonal of the larger indentation.

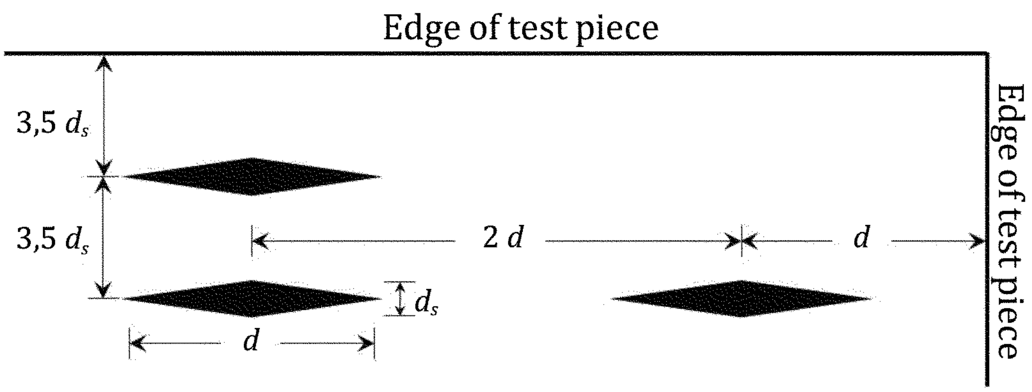


**Figure 3 — Minimum distance for Knoop indentations**

 

## 8.9 Measurement of diagonal length

The length of the long diagonal shall be measured and used for the calculation of the Knoop hardness. For all tests, the perimeter of the indentation shall be clearly defined in the field of view of the microscope.

Magnifications should be selected so that the diagonal can be enlarged to greater than 25 %, but less than 75 % of the maximum possible optical field of view (see 6.3).

NOTE 1 In general, decreasing the test force increases the scatter of the results of the measurements. The accuracy of the determination of the long diagonal length is unlikely to be better than ±0,001 mm.

NOTE 2 A helpful technique for adjusting optical systems that have Köhler illumination is given in Annex E.

If the shape of the indentation appears to be nonsymmetrical, divide the long diagonal into two segments at the point of intersection with the short diagonal, and measure the length of each segment. If the difference between the two segments is greater than 5 % of the length of the long diagonal, check the parallelism between the supporting plane and the measuring plane of the specimen and eventually, the alignment of the indenter to the specimen. Test results with deviations greater than 5 % should be discarded.

## 8.10 Calculation of hardness value

Calculate the Knoop hardness value using the formula given in Table 1. The Knoop hardness value can also be determined using the calculation tables given in ISO 4545-4.

# 9 Uncertainty of the results

A complete evaluation of the uncertainty should be done according to JCGM 100: 2008[7].

Independent of the type of sources, for hardness, there are two possibilities for the determination of the uncertainty.

— One possibility is based on the evaluation of all relevant sources appearing during a direct calibration. As a reference, a Euramet guideline[8] is available.

— The other possibility is based on indirect calibration using a hardness reference block [below abbreviated as CRM (certified reference material)] (see References [8] to [11]). A guideline for the determination is given in Annex B.

It may not always be possible to quantify all the identified contributions to the uncertainty. In this case, an estimate of type A standard uncertainty may be obtained from the statistical analysis of repeated indentations into the test piece. Care should be taken if standard uncertainties of type A and B are summarized, that the contributions are not counted twice (see JCGM 100: 2008, Clause 4).

# 10 Test report

The test report shall include the following information, unless otherwise agreed by the parties concerned:

a) a reference to this document, i.e. ISO 4545-1;

b) all information necessary for identification of the test piece;

c) the date of the test;

d) the hardness result obtained in HK, reported in the format defined in 5.2;

e) all operations not specified in this document, or regarded as optional;

f) the details of any circumstances that affected the results;

g) the temperature of the test, if it is outside the ambient range specified in 8.1;

h) where conversion to another hardness scale is also performed, the basis and method of this conversion.

There is no general process of accurately converting Knoop hardness values into other scales of hardness or into tensile strength. Such conversions, therefore, should be avoided, unless a reliable basis for conversion can be obtained by comparison tests (see also ISO 18265).

NOTE A strict comparison of hardness values is only possible at identical test forces.



 

# Annex A
(normative)

# Procedure for periodic checking of the testing machine, diagonal measuring system and the indenter by the user

## A.1 Periodic verification

The indenter to be used for the periodic verification shall be the same as used for testing. A hardness reference block shall be chosen for testing that is calibrated in accordance to ISO 4545-3 on the scale and at the approximate hardness level at which the machine will be used.

Before performing the periodic verification, the diagonal measuring system shall be indirectly verified using one of the reference indentations on the hardness reference block. The measured indentation length shall agree with the certified value to within the greater of 0,001 mm or 1,25 % of the indentation length. If the diagonal measuring system fails this test, a second reference indentation may be measured. If the diagonal measuring system fails this test a second time, the diagonal measuring system shall be adjusted or repaired and undergo direct and indirect verification according to ISO 4545-2.

At least two hardness measurements shall be made on the calibrated surface of the hardness reference block. The indentations shall be uniformly distributed over the surface of the reference block. The machine is regarded as satisfactory if the maximum positive or negative percent bias, $b_{rel}$ , for each reading does not exceed the limits shown in Table A.1.

The percent bias, $b_{rel}$, is calculated according to Formula (A.1):

$$b_{rel} = 100 \times \frac{H - H_{CRM}}{H_{CRM}} \quad \text{(A.1)}$$

where

$H$ is the hardness value corresponding to the hardness measurement taken;

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

If the testing machine fails this test, verify that the indenter and testing machine are in good working condition and repeat the periodic verification. If the machine continues to fail the periodic verification, an indirect verification according to ISO 4545-2 shall be performed. A record of the periodic verification results should be maintained over a period of time and used to measure reproducibility and monitor drift of the machine.

**Table A.1 — Maximum permissible percent HK bias**

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

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 periodic verification of the machine.

## A.2 Indenter inspection

Experience has shown that a number of initially satisfactory indenters can become defective after use for a comparatively short time. This is due to small cracks, pits or other flaws in the surface. If such faults are detected in time, many indenters may be reclaimed by regrinding. If not, any small defects on the surface rapidly worsen and make the indenter useless. Therefore,

— the condition of indenters should be monitored by visually checking the aspect of the indentation on a reference block, each day the testing machine is used,

— the verification of the indenter is no longer valid when the indenter shows defects, and

— reground or otherwise repaired indenters shall meet all of the requirements of ISO 4545-2.

 

# Annex B
(informative)

# Uncertainty of the measured hardness values

## B.1 General requirements

Measurement uncertainty analysis is a useful tool to help determine sources of error and to understand differences in test results. This annex gives guidance on uncertainty estimation but the methods contained are for information only, unless specifically instructed otherwise by the customer.

Most product specifications have tolerances that have been developed over the past years based mainly on the requirements of the product but also, in part, on the performance of the machine used to make the hardness measurement. These tolerances therefore incorporate a contribution due to the uncertainty of the hardness measurement and it would be inappropriate to make any further allowance for this uncertainty by, for example, reducing the specified tolerance by the estimated uncertainty of the hardness measurement. In other words, where a product specification states that the hardness of an item shall be higher or lower than a certain value, this should be interpreted as simply specifying that the calculated hardness value(s) shall meet this requirement, unless specifically stated otherwise in the product standard. 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 approach for determining uncertainty presented in this annex considers only those uncertainties associated with the overall measurement performance of the hardness testing machine with respect to the hardness reference blocks (abbreviated as CRM below). These performance uncertainties reflect the combined effect to all the separate uncertainties (indirect verification). Because of this approach, it is important that the individual machine components are operating within the tolerances. It is strongly recommended that this procedure be applied for a maximum of one year after the successful passing of a direct verification.

Annex C shows the four-level structure of the metrological chain necessary to define and disseminate hardness scales. The chain starts at the international level using international definitions of the various hardness scales to carry out international intercomparisons. A number of primary hardness standard machines at the national level "produce" primary hardness-reference blocks for the calibration laboratory level. Naturally, direct calibration and the verification of these machines should be at the highest possible accuracy.

## B.2 General procedure

The procedure calculates a combined uncertainty, $u$, by the root-squared-sum-method (RSS) out of the different sources given in Table B.1. The expanded uncertainty, $U$, is derived from $u$ by multiplying with the coverage factor $k = 2$. Table B.1 contains all symbols and their designation.

The bias, $b$, of a hardness testing machine (also named "error"), which is derived from the difference between

— the certified calibration value of the hardness reference block used, and

— the mean hardness value of the five indentations made in this block during calibration of the hardness testing machine,

can be implemented in different ways into the determination of uncertainty (see ISO 4545-2).

Two methods are given for determining the uncertainty of hardness measurements.

— Method 1 (M1): accounts for the systematic bias of the hardness machine in two different ways. In one approach, the uncertainty contribution from the systematic bias is added arithmetically to this value. In the other approach, a correction is made to the measurement result to compensate for the systematic bias.

— Method 2 (M2): allows the determination of uncertainty without having to consider the magnitude of the systematic bias.

Additional information on calculating hardness uncertainties can be found in the literature (see References [7] and [8]).

NOTE 1 This uncertainty approach makes no allowance for any possible drift in the machine performance subsequent to its last calibration, as it assumes that any such changes will be insignificant in magnitude. As such, most of this analysis could be performed immediately after the machine's calibration and the results included in the machine's calibration certificate.

NOTE 2 In this annex, the abbreviation "CRM" stands for "certified reference material". In hardness testing standards, certified reference material is equivalent to the hardness reference block, i.e. a piece of material with a certified value and associated uncertainty.

## B.3 Procedures for calculating uncertainty — Hardness measurement values

### B.3.1 Procedure with bias (method M1)

The method M1 procedure for the determination of measurement uncertainty is explained in Table B.1. The measurement bias, $b$, of the hardness testing machine can be expected to be a systematic effect. In JCGM 100: 2008, it is recommended that a correction be used to compensate for systematic effects, and this is the basis of M1. The result of using this method is that either all determined hardness values $x$ have to be reduced by $b$ or the uncertainty, $U$, has to be increased by $b$. The procedure for the determination of $U_{M1}$ is explained in Table B.1.

The combined expanded measurement uncertainty for a single hardness measurement, $x$, is calculated according to Formula (B.1):

$$U_{M1} = k \times \sqrt{u_H^2 + 2 \times u_{ms}^2 + u_{HTM}^2} \quad \text{(B.1)}$$

where

$u_H$ is a contribution to the measurement uncertainty due to the lack of measurement repeatability of the hardness testing machine;

$u_{ms}$ is a contribution to the measurement uncertainty due to the resolution 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 $u_H$ due to resolving the positions of both ends of the long diagonal independently;

$u_{HTM}$ is a contribution to the measurement uncertainty due to the standard uncertainty of the bias measurement, $b$, generated by the hardness testing machine (this value is reported as a result of the indirect verification defined in ISO 4545-2) and is calculated according to Formula (B.2):

$$u_{HTM} = \sqrt{u_{CRM}^2 + u_{HCRM}^2 + 2 \times u_{ms}^2} \quad \text{(B.2)}$$

 

where

$u_{CRM}$ is the contribution to the measurement uncertainty due to the calibration uncertainty of the certified value of the CRM according to the calibration certificate for $k = 1$;

$u_{HCRM}$ is the contribution to the measurement uncertainty due to the combination of the lack of measurement repeatability of the hardness testing machine and the hardness nonuniformity of the CRM, calculated as the standard deviation of the mean of the hardness measurements when measuring the CRM;

$u_{ms}$ is the contribution to the measurement uncertainty due to the resolution of the hardness testing machine when measuring the CRM.

The result of the measurement can be reported in two ways:

— as $X_{corr}$, where the measurement value, $x$, is corrected for the measurement bias, $b$, calculated according to Formula (B.3):

$$X_{corr} = (x - b) \pm U_{M1} \quad \text{(B.3)}$$

— as $X_{ucorr}$, where the measurement value, $x$, is not corrected for the measurement bias, $b$, and the expanded uncertainty, $U$, is increased by the absolute value of the bias calculated according to Formula (B.4):

$$X_{ucorr} = x \pm \left[ U_{M1} + |b| \right] \quad \text{(B.4)}$$

When method M1 is used, it can also be appropriate to include additional uncertainty contributions within the RSS term relating to the value of $b$ employed. This will particularly be the case when

— the measured hardness is significantly different from the hardness levels of the blocks used during the machine's calibration,

— the machine's bias value varies significantly throughout its calibrated range,

— the material being measured is different from the material of the hardness reference blocks used during the machine's calibration, or

— the day-to-day performance (reproducibility) of the hardness testing machine varies significantly.

The calculations of these additional contributions to the measurement uncertainty are not discussed here. In all circumstances, a robust method for estimating the uncertainty associated with $b$ is required.

### B.3.2 Procedure without bias (method M2)

As an alternative to method M1, method M2 can be used in some circumstances. Method M2 is only valid for hardness testing machines that have passed an indirect verification in accordance with ISO 4545-2 using the value $|b| + U_{HTM}$, rather than only the bias value, $b$, when determining compliance with the maximum permissible deviation of the bias (see ISO 4545-2). In method M2, the maximum permissible bias, $b_E$ (the positive amount by which the machine's reading is allowed to differ from the reference block's value), as specified in ISO 4545-2:2017, Table 2, is used to define one component, $u_E$, of the uncertainty. There is no correction of the hardness values with respect to the bias limit. The procedure for the determination of $U$ is explained in Table B.1.

 

The combined expanded measurement uncertainty for a single future hardness measurement is calculated according to Formula (B.5):

$$U_{M2} = k \times \sqrt{u_H^2 + 2 \times u_{ms}^2 + u_E^2} \qquad \text{(B.5)}$$

where

$u_H$ is a contribution to the measurement uncertainty due to the lack of measurement repeatability of the hardness testing machine;

$u_{ms}$ is a contribution to the measurement uncertainty due to the resolution 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 $u_H$ due to resolving the positions of both ends of the long diagonal independently;

$u_E$ is a contribution to the measurement uncertainty due to the maximum permissible deviation of the bias, (rectangular distribution), where $b_E$ is the maximum permissible deviation of the bias as specified in ISO 4545-2, and the result of the measurement is calculated according to Formula (B.6):

$$X = \mathrm{x} \pm U_{M2} \qquad \text{(B.6)}$$

## B.4 Expression of the result of measurement

EXAMPLE A hardness testing machine makes a single Knoop hardness measurement, *x*, on a test sample.

| | |
|---|---|
| Single hardness measurement value, *x*: | *x* = 810 HK 1 |
| Diagonal length, *d*: | *d* = 0,132 5 mm |

Resolution of the length diagonal measuring system is calculated according to Formula (B.7):

$$\delta_{ms} = \sqrt{\delta_{OR}^2 + \delta_{IR}^2} \qquad \text{(B.7)}$$

$\delta_{ms}$ = 0,000 51 mm

where

$\delta_{OR}$ is the optical resolution of the microscope objective (0,000 5 mm);

$\delta_{IR}$ is the resolution of the display indicator of the measuring system (0,000 1 mm).

The last indirect verification of the testing machine determined a measurement bias, *b*, with an uncertainty of the bias, $U_{HTM}$, using a CRM of $\overline{H}_{CRM}$ = 802,7 HK 1. The hardness of this CRM was the closest to the test sample hardness of those CRMs used for the indirect verification.

| | |
|---|---|
| Testing machine measurement bias, *b*: | *b* = 1,0 HK 1 |
| Uncertainty of the testing machine measurement bias, $U_{HTM}$: | $U_{HTM}$ = 12,7 HK 1 |

To determine the lack of repeatability of the testing machine, the laboratory made five HK 1 measurements, $H_i$, on a CRM having a similar hardness to the test sample. The five measurements were made adjacent to each other adhering to spacing requirements in order to reduce the influence of block non-uniformity.

Five measurement values, $H_i$: 806,5 HK 1; 803,0 HK 1; 800,9 HK 1; 803,4 HK 1; 797,5 HK 1

 

Mean measurement value, $\overline{H}$:   $\overline{H}$ = 802,3 HK 1

Standard deviation of the measurement values, $s_H$:   $s_H = 3{,}3\ \mathrm{HK\,1}$

The value of $s_H$ based on measurements from the last indirect verification according to ISO 4545-2 may be used instead of conducting the above repeatability tests; however, this standard deviation value will usually overestimate the lack of repeatability uncertainty component since it also includes the CRM non-uniformity.

For this example,

$$|b| + U_{HTM} = 1{,}0 + 12{,}7 = 13{,}7\ \mathrm{HK\,1}$$, and

$b_E$ = 4 % of 810 HK 1 = 32,4 HK 1.

Since the testing machine bias plus the expanded uncertainty in determining the bias, $\left[|b| + U_{HTM}\right]$, is within the maximum permissible bias, $b_E$, either Method 1 or Method 2 may be used.

 

**Table B.1 — Determination of the expanded uncertainty according to methods M1 and M2**

| Step | Sources of uncertainty | Symbols | Formula | Literature/Certificate | Example |
|---|---|---|---|---|---|
| **1** **M1,M2** | Measurement result | $x$ | | | $x$ = 810 HK 1 |
| **2** **M1** | Bias value, $b$, and uncertainty, $U_{HTM}$, of the bias of the hardness testing machine from the indirect verification | $b$ $U_{HTM}$ $u_{HTM}$ | $u_{HTM} = \frac{U_{HTM}}{2}$ | $b$ and $U_{HTM}$ according to indirect verification report using a CRM of $\bar{H}_{CRM} = 802{,}7 \text{HK1}$ (see Note 1) | $b$ = 1,0 HK 1 $U_{HTM}$ = 12,7 HK 1 $u_{HTM} = \frac{12{,}7}{2} = 6{,}35 \text{HK1}$ |
| **3** **M2** | Maximum permissible deviation of the bias | $b_E$ | $b_E$ = Maximum positive value of permissible bias | Permissible bias, $b$ according to ISO 4545-2:2017, Table 3 | $b_E$ = 4 % $b_E = \frac{4 \times 810}{100}$ = 32,4 HK 1 |
| **4** **M2** | Standard uncertainty due to the maximum permissible deviation of the bias | $u_E$ | $u_E = b_E / \sqrt{3}$ | Rectangular distribution | $u_E = \frac{32{,}4}{\sqrt{3}}$ = 18,7 HK 1 |
| **5** **M1,M2** | The standard deviation of repeatability measurements | $s_H$ | $s_H = \sqrt{\frac{1}{n-1} \sum_{i=1}^{n} \left(H_i - \bar{H}\right)^2}$ | Five measurements are made by the laboratory on a CRM having a hardness similar to the test sample (see Note 2) | $s_H$ = 3,3 HK 1 |
| **6** **M1,M2** | Standard uncertainty due to lack of repeatability | $u_H$ | $u_H = t \times s_H$ | $t$ = 1,14 for $n$ = 5 (see JCGM 100:2008)[5] | $u_H$ = 1,14 × 3,3 = 3,8 HK 1 |

**Table B.1** *(continued)*

| Step | Sources of uncertainty | Symbols | Formula | Literature/Certificate | Example |
|---|---|---|---|---|---|
| **7** **M1,M2** | Standard uncertainty due to resolution of the hardness value indicating display | $u_{ms}$ | $u_{ms}=-\frac{2x}{d}\times\frac{\delta_{ms}}{2\sqrt{3}}$ | $\delta_{ms}$ = 0,000 51 mm<br>$x$ = 810 HK 1<br>$d$ = 0,133 mm<br>(see Note 3) | $u_{ms}=-\frac{2\times 810{,}0}{0{,}133}\times\frac{0{,}00051}{2\times\sqrt{3}}=-1{,}80\text{HK}1$ |
| **8** **M1** | Determination of the expanded uncertainty | $U_{M1}$ | $U_{M1}=k\times\sqrt{u_H^2+2\times u_{ms}^2+u_{HTM}^2}$ | Steps 2, 6, and 7<br>$k$ = 2 | $U_{M1}$ = 15,6 HK 1 |
| **9** **M1** | Measurement result with modified hardness | $X_{corr}$ | $X_{corr}=(x-b)\pm U_{M1}$ | Steps 1, 2 and 8 | $x$ = 810 HK 1<br>$X_{corr}$ = (809 ± 16) HK 1 |
| **10** **M1** | Measurement result with modified uncertainty | $X_{ucorr}$ | $X_{ucorr}=x\pm(U_{M1}+\lvert b\rvert)$ | Steps 1, 2 and 8 | $x$ = 810 HK 1<br>$X_{ucorr}$ = (810 ± 17) HK 1 |
| **11** **M2** | Determination of the expanded uncertainty | $U_{M2}$ | $U_{M2}=k\times\sqrt{u_H^2+2\times u_{ms}^2+u_E^2}$ | Steps 4, 6, and 7<br>$k$ = 2 | $U_{M2}$ = 38,5 HK 1 |
| **12** **M2** | Measurement result | $X$ | $X=x\pm U_{M2}$ | Steps 1 and 11 | $x$ = 810 HK 1<br>$X$ = (810 ± 39) HK 1 |

NOTE 1 If 0,8 $b_E < b <$ 1,0 $b_E$, the relationship of hardness values between CRM and sample can be considered.

NOTE 2 The value of $s_H$ based on measurements from the last indirect verification according to ISO 4545-2 can be used, but will usually overestimate the lack of repeatability uncertainty component since it includes the CRM non-uniformity. In circumstances where the average of multiple hardness measurements on a test sample is to be reported, rather than a single hardness measurement, the value of $s_H$ in Step 5 should be replaced with the standard deviation of the multiple hardness measurements of the sample under test divided by the square-root of the number of hardness measurements, $n$, and the value of $t$ should be appropriate for the $n$ measurements ($u_H=t\times s_H/\sqrt{n}$). The calculated uncertainty contribution, $u_H$, will then also account for the nonuniformity of the test sample.

NOTE 3 The sensitivity coefficient, $-2x/d$, follows from $\partial x/\partial d$ for converting uncertainty in diagonal length (mm) to uncertainty in HK.

# Annex C
(informative)

# Knoop hardness measurement traceability

## C.1 Traceability definition

The path to traceability for a Knoop hardness measurement is different compared to many other measurement quantities, such as length or temperature. This is primarily because hardness measurement including Knoop is made following a defined test procedure using a testing machine that makes multiple measurements of different parameters (e.g. force, length, time) during the test. Each of these measurements, as well as other test parameters, influences the hardness result.

The International Vocabulary of Metrology (VIM3)[12] defines metrological traceability as:

> ***metrological traceability*** — *property of a measurement result whereby the result can be related to a reference through a documented unbroken chain of calibrations, each contributing to the measurement uncertainty, VIM3, 2012.*

From this definition, two things are necessary for a measurement result to have traceability:

1) an unbroken chain of calibrations, each contributing to the measurement uncertainty;

2) a reference to which traceability is claimed.

These will define the metrological traceability chain.

## C.2 Chains of calibrations

ISO 4545-2 specifies a set of calibration and verification procedures required to demonstrate that the testing machine is suitable for use in accordance with this document. The calibration procedures include direct measurements of various components affecting the machine's performance — such as the test forces, indenter shape, and diagonal measuring equipment — as well as hardness measurements of a range of reference blocks. Each of these calibration measurements has specified limits within which the result shall lie in order for the machine to pass its verification. Historically, the calibration and verification of the machine components has been termed the machine's direct verification and the calibration and verification of the testing machine by reference block measurements its indirect verification.

ISO 4545-3 specifies both the procedure required to calibrate the reference blocks used in the indirect verification of the testing machine and also the required calibration and verification procedures of the machine used to calibrate these blocks.

When considering an "unbroken chain of calibrations" to provide measurement traceability to the testing machine, it is apparent that this could come via either the direct verification or indirect verification path.

Direct verification requirements specify measurements of individual components of the testing machine, with traceability of each of these measurements being achieved through calibration chains to the International System of Units (SI), usually as realized by a National Metrology Institute (NMI). These calibration chains are illustrated on the right-hand side of Figure C.1. Together, these calibration chains form a potential traceability path for a testing machine.

The left-hand side of Figure C.1 illustrates a traceability path made through a single calibration chain for each level in the calibration hierarchy (i.e. national, calibration and user) that includes the calibration of reference blocks and the subsequent indirect verification of Knoop hardness machines. A (national level) primary standard machine calibrates primary reference blocks that are then used to calibrate a (calibration level) calibration machine. This machine calibrates reference blocks that are finally used to calibrate a (user level) testing machine.

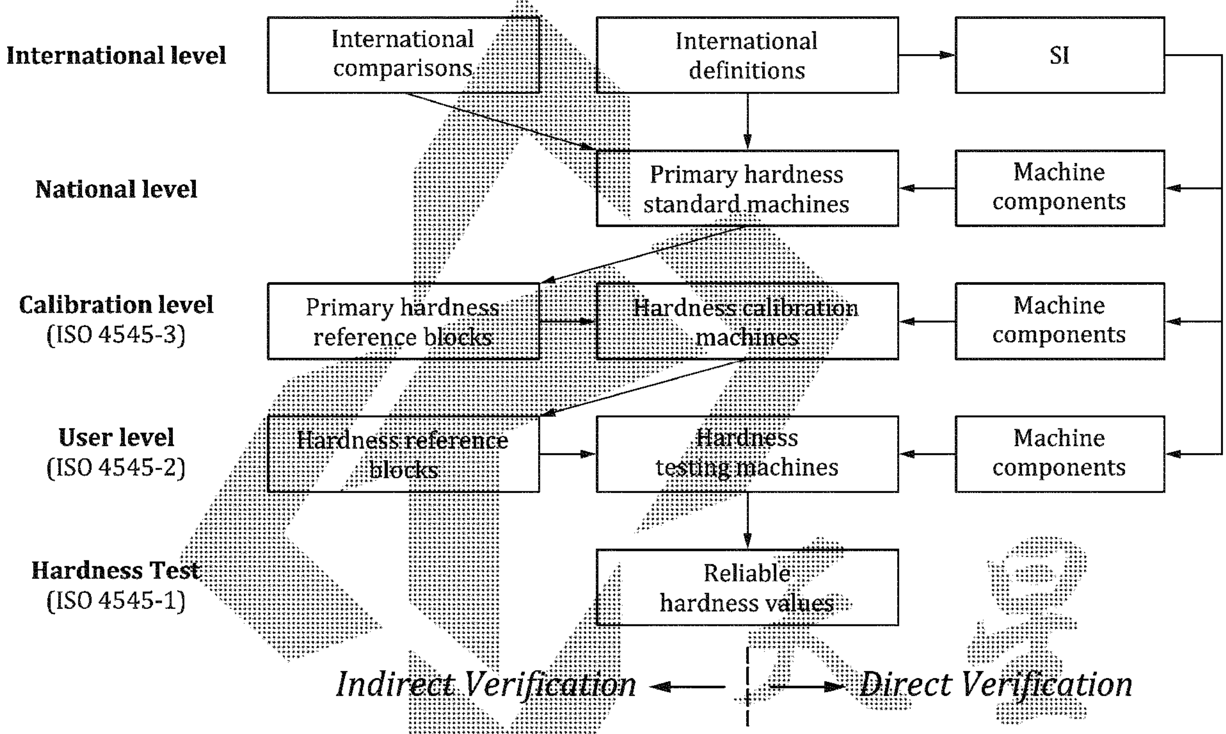


**Figure C.1 — Chains of calibrations**

## C.3 Knoop hardness reference

The other requirement for achieving traceability is a reference to which traceability is claimed. Knoop hardness is not a fundamental property of a material, but rather an ordinal quantity dependent on a defined test method. Ideally, the ultimate reference for a Knoop hardness measurement should be an internationally agreed definition of this method, including values of all test parameters. Hardness traceability would then be to this definition through a laboratory's realization or fulfillment of the definition, the accuracy of this realization being reflected in the laboratory's measurement uncertainty and confirmed by international comparisons. The internationally agreed definition would be developed by the CCM working group on hardness (CCM-WGH) (see Annex D) and realized by NMIs that standardize Knoop hardness. At this time, the CCM-WGH has not developed definitions for Knoop hardness scales so the highest reference is usually an NMI's realization of the Knoop scales based on its own chosen definition of the test. In cases where an NMI does not calibrate reference blocks for certain Knoop scales, the highest reference within a country may be a calibration laboratory's realization of the Knoop scale definition.

## C.4 Practical issues

Either one of the two traceability paths of calibration chains illustrated in Figure C.1 (left-hand side and right-hand side) could theoretically provide traceability to an appropriate Knoop hardness reference. However, there are practical issues with both that shall be considered. For the direct verification path given on the right-hand side of Figure C.1, it is extremely difficult to identify, measure, and if necessary, correct for all parameters that may affect the measured hardness value. Even if the machine passes its direct verification, traceability will not be assured if one or more uncontrolled or unidentified parameters have a significant effect. This is often the case and becomes more of an issue at lower levels in the calibration hierarchy.

 

The indirect verification calibration chain shown on the left-hand side of Figure C.1 also has practical issues to be considered. One consequence of using a testing machine having multiple components, each making measurements during the hardness test, is that an error in one component's measurement can be compensated or offset by an error in a different component's measurement. This can result in accurate hardness measurements for the specific hardness levels and block materials tested during the indirect verification; however, measurement error can increase when testing other hardness levels or materials. If the errors in the individual machine components are significant, then traceability again may not be assured.

## C.5 Knoop hardness measurement traceability

### C.5.1 General

The above issues indicate that both types of traceability path usually need to be in place for achieving Knoop hardness measurement traceability. However, traceability can be achieved based on only one of the two paths if careful examination and evaluation of the measurement process is made. For example, at the national level, the traceability of an NMI's primary Knoop hardness standard machine is achieved through a direct verification calibration chain since there is no recognized higher-level hardness reference artifact. Traceability through this path is possible since NMIs typically have the capability to thoroughly evaluate their measurement systems, and their uncertainty levels are confirmed through international comparisons with other NMIs. In contrast, decades of Knoop hardness measurement experience has shown that, for the lower levels in the calibration hierarchy, it is most practical to obtain traceability and determine measurement uncertainty based primarily on the indirect verification calibration chain; however, proper traceability of the individual machine component quantity values is also important. This traceability scheme has proven to be suitable for industrial Knoop hardness measurements.

### C.5.2 Calibration level traceability

Measurement traceability is best obtained through the indirect verification calibration chain using primary reference blocks that have been calibrated at the national NMI level. This is also the path that should be used for the determination of measurement uncertainty. At the same time, however, the specified components of the calibration machine should be calibrated on a frequent basis to ensure that offsetting errors are not significant. Hardness traceability should be to the NMI's realization of the CCM-WGH definition of the Knoop scale or, when there is an absence of a CCM-WGH definition; traceability should be to the NMI's realization of its own chosen definition. If the NMI does not provide calibrated reference blocks or conduct comparison measurements with a calibration laboratory and it is not practical to use reference blocks of another NMI, then the reference to which traceability is claimed may need to be to the calibration laboratory's realization of the Knoop scale definition based on an international test method, such as that defined by this document. In this case, the calibration laboratory's measurement traceability may be achieved through the indirect verification path using consensus reference block standards, or through the direct verification path confirmed by intercomparisons.

### C.5.3 User level traceability

Measurement traceability is best obtained through the indirect verification calibration chain using reference blocks that have been calibrated at the calibration level or national level. As with calibration level traceability, this is the most practical path and should also be used for the determination of measurement uncertainty. It is also desirable that the components of the hardness machine periodically undergo direct verification to ensure that offsetting errors are not significant. However, typical industrial practice is for these measurements to be made only when the hardness machine is manufactured or repaired, which is the minimum requirement of this document.

 

NOTE The following terms used in this annex are in accordance with the VIM3:

— calibration;
— calibration hierarchy;
— metrological traceability;
— metrological traceability chain;
— ordinal quantity;
— verification.

# Annex D
(informative)

# CCM — Working group on hardness

In 1999, at the 88th Session of the International Committee of Weights and Measures (CIPM), Dr Kozo Iizuka, President of the Consultative Committee for Mass and Related Quantities (CCM), stated "Although the definition of hardness scales is certainly conventional in the sense of the use of arbitrarily chosen formula, the testing method is defined by a combination of physical quantities expressed by SI units; the standard of hardness is established and maintained in most of NMIs and the traceability to the standard of NMIs is demanded in industry and elsewhere." The subsequent discussions led to the realization that hardness standards should be included in the key comparison database (KCDB) for the Mutual Recognition Arrangement (MRA), and thus a full working group on hardness (CCM-WGH) was established in the framework of the CCM[13].

The establishment of the CCM-WGH provided a technical-diplomatic framework in which hardness influence parameters can be examined, and improved international definitions of the hardness tests can be proposed and approved for NMI use to reduce the measurement differences at the highest national level. Due to the necessity of international agreement, the CCM-WGH has a close liaison with ISO/TC 164/SC 3 in order to assure proper dissemination of the hardness scales. The most significant improvement of the CCM-WGH definitions is that the parameters of the hardness test are defined with specific values, rather than ranges of acceptable limits as specified by this test method. As applicable, this document has adopted the defined values of the CCM-WGH definitions as the values to use.

The CCM-WGH definitions are published at http://www.bipm.org/.

 

# Annex E
(informative)

# Adjustment of Köhler illumination systems

## E.1 General

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

## E.2 Köhler 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 the 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.

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