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Traceability of hardness measurements

Contents

Changes since last edition

- Term “should” updated to “shall” where a requirement is mandated within the relevant hardness standard(s)
- Information provided relating to bias and uncertainty
- General review and clarification throughout
- Section 13 ‘Reference standards’ added

1. Introduction

1.1 Laboratories that have been assessed by UKAS as meeting the requirements of ISO/IEC 17025 *General requirements for the Competence of Testing and Calibration Laboratories* may be granted UKAS Accreditation. Several guidance publications on the application of these requirements, providing extra information, detail and limitations are listed in the publications section of the UKAS website.

1.2 Calibration and verification of hardness testing equipment is required to be traceable to national standards. When using external calibration sources, a valid certificate shall be obtained from a calibration laboratory that demonstrates competence, measurement capability and traceability. A calibration certificate bearing the UKAS accreditation mark (or identity of the national standards laboratory or mark of an accreditation body with which UKAS has a recognition agreement) for the relevant calibration/verification will be sufficient evidence.

1.3 This publication provides guidance for laboratories needing to ensure traceability of hardness measurements. By following this guidance, laboratories will be able to demonstrate that they meet the requirements of ISO/IEC 17025 and the relevant hardness testing standards. Alternative methods may be used provided they are shown to give an equivalent outcome.

2. Direct verification

2.1 All hardness machines shall be directly verified as evidenced by a calibration certificate (see 1.2 above). The certificate shall report the location of the machine, all the measured values and the estimated uncertainty of the measured values. The direct verification shall be performed using the customer's indenters.

2.2 Intervals between direct verifications

a) Calibration machines shall be directly verified at intervals no greater than twelve months
b) Testing machines shall be directly verified at first installation
c) Testing machines shall be directly verified following failure of an indirect verification or if the maximum permitted interval since the last indirect verification has been exceeded (as specified by the relevant standard)
d) Testing machines should be directly verified after dismantling and reassembling if there is a risk that the force, measuring system or test cycle could have been affected
e) Testing machines should be directly verified if moved from the location where the previous direct verification was performed
f) Relocation of a machine should be performed by a qualified service and calibration engineer
g) Each indenter should be directly verified every two years

3. Indirect verification

3.1 An indirect verification is only valid for a machine that has a valid direct verification. All hardness machines shall be indirectly verified and this evidenced by a calibration certificate (see 1.2 above) for both direct and indirect verification. The certificate shall report the location of the machine. It shall also report all the measured hardness values, and the estimated uncertainty of the measured hardness values obtained in the indirect verification. The certificate shall clearly identify the hardness machine, the indenter(s) and the reference block(s) used in the verification. The indirect verification shall be performed using the customer's indenters and for each indenter that the customer will use.

3.2. Intervals between indirect verifications

a) Calibration and testing machines shall be indirectly verified following a direct verification
b) Calibration and testing machines shall be indirectly verified at least once every twelve months
c) Testing machines shall be indirectly verified after dismantling and reassembling if there is a risk that the force, measuring system or test cycle could have been affected
d) Testing machines shall be indirectly verified if moved from the location where the previous direct verification was performed

4. Traceability of reference blocks

4.1 All primary hardness reference blocks used for the verification of hardness calibration machines shall be calibrated as evidenced by a valid calibration certificate. The certificate shall report all the measured values and the estimated uncertainty of the measured values.

4.2 Certification of primary hardness reference blocks shall be obtained from a National Measurement Institute that is a signatory of the S.I. mutual recognition agreement and which has published in the BIPM key comparison database a CMC for realisation of the relevant hardness scale. For example:

a) **Istituto Nazionale di Ricerca Metrologica** (INRIM), Italy
b) or **Physikalisch-Technische Bundesanstalt** (PTB), Germany
c) or **National Institute of Standards and Technology** (NIST), USA

4.3 Reference blocks used for the verification of hardness testing machines shall be calibrated and this shall be evidenced by a valid calibration certificate obtained from an ISO/IEC 17025 accredited calibration laboratory (see 1.2 above). The certificate shall report all the measured values and the estimated uncertainty of the measured values.

4.4 Reference blocks used for regular checks shall be calibrated as evidenced by a valid calibration certificate obtained from an ISO/IEC 17025 accredited calibration laboratory (see 1.2 above). The certificate shall report all the measured values and the estimated uncertainty of the measured values.

4.5 Reference blocks for Vickers, Knoop and Brinell Testing shall contain a reference indentation. This shall be used to carry out indirect verification and regular monitoring of the optical measuring equipment.

4.6 All reference blocks shall be suitably protected, maintained in a satisfactory condition in accordance with the requirements of the relevant hardness standard and ISO/IEC 17025 and should be used solely for the purpose of hardness measurement.

4.7 A permanent record shall be kept of the results of all indirect verifications and periodic checks performed. A control chart should be constructed for each reference hardness block to monitor the reproducibility of a machine and indenter combination over time.

5. Traceability of diamond hardness indenters

5.1 All diamond indenters for Vickers, Knoop, Rockwell and Rockwell Superficial shall be directly verified as evidenced by a valid calibration certificate (see 1.2 above). The certificate shall report all the measured values and the estimated uncertainty of the measured values. Each indenter should be directly verified every two years.

6. Traceability of ball indenters

6.1 All ball indenters for Rockwell and Brinell shall be verified. The certificate should report all the measured values and the estimated uncertainty of the measured values.

6.2 The relevant standards specify sampling requirements and limits for ball diameter; roundness; surface finish; hardness; chemical composition and density.

6.3 Indenter balls should be obtained from a bonded batch and evidenced by a valid certificate confirming compliance with the relevant standard specifications.

6.4 All calibration certificates should contain actual measured values together with the estimated uncertainty of that measurement.

7. Indentation measuring equipment

7.1 The indirect verification of integral indentation measuring equipment is carried out as part of the indirect verification of a hardness machine. If auxiliary measuring equipment (such as hand microscopes, scanning systems and profile projectors) is used, then it shall be subjected to the same calibrations and verifications as integral measuring equipment. In all cases the measuring system shall be calibrated according to the relevant section of the hardness standard for: every objective lens, all line scales used, and in all measurement directions used. The verification shall be evidenced by a valid calibration certificate (see 1.2 above).

7.2 To ensure that the lighting, numerical aperture of the lens and operator discrimination meet requirements, readings shall be obtained from the reference indentations of the reference hardness blocks used for the indirect verification. The readings obtained shall be within the permitted tolerance given in the relevant standard.

7.3 If auxiliary measuring devices fail indirect verification the device shall be directly calibrated and verified or replaced by a device that passes direct verification (and the associated required indirect verification).

7.4 Integral and auxiliary measuring devices used for the measurement of Brinell indentations can be classified into two distinct types:

1: Devices that include microscopes capable of selecting measurement points in an electronic field of view (these may have a cursor or moveable measuring lines) that rely upon a computerised measuring system or an image analysis system.
Note: In systems that measure point to point in any direction, calibration of the measuring device in two orthogonal directions is required. In systems using lines to select a measuring position, the alignment of the measured distance with the axis of calibration is essential.

2: Integral or handheld microscopes with fixed measuring lines such as an eyepiece graticule.

When working to ASTM E10 type 2 handheld microscopes with fixed measuring lines shall not be used for measuring indentations made with 2,5 mm or 1 mm ball indenters.

When working to ISO 6506, handheld microscopes with fixed measuring lines are not graduated with sufficient resolution to permit estimation of the indent diameter to within 0.5% for indentations < 4 mm using a x 20 magnification or < 2 mm using a x 40 magnification.

8. Regular monitoring by in-house checks

8.1 Laboratories holding accreditation for hardness testing shall carry out regular in-house checks using reference blocks, which meet the requirements of Section 4.

8.2 In-house checks shall be made at time intervals specified by the testing standard. If no time period is specified, checks should be made before use on each day the machine is used. The checks should be made under conditions of force and hardness (i.e. indent size) comparable to those to be generated in the subsequent hardness test measurements. Permanent records shall be made of each check. A machine shall be deemed satisfactory if all hardness readings fall within the tolerance values for monitoring hardness machines as stated in the relevant standard specification. If not, an indirect verification shall be performed.

8.3 Measuring devices including microscopes and scanning systems should be checked before the hardness check in paragraph 8.1 or as required by the relevant standard specification. This shall be done by measuring a reference indentation on a reference block using the same lighting conditions as used for the last indirect validation of the machine and where the reference indentation is of a similar size to the indentations on the work to be tested. It is recommended that the reference indentation also exhibit a similar amount of pile up as the material to be tested. Permanent records shall be made of each check.

9. Portable hardness testers and comparators

9.1 In cases where it is possible to establish traceability directly and indirectly to verify portable hardness testers, then this shall be carried out in line with the relevant standards.

9.2 Where portable hardness testers are directly and indirectly verified each removable part (loadcell, measuring system etc.) shall have a serial number which shall be referenced on the calibration certificate. The calibration certificate is only valid for a system with the exact combination of removable parts used for the calibration.

9.3 In cases where no route exists to establish the traceability of verification of portable hardness testing machines, accreditation is limited to comparative testing.

9.4 Portable hardness testing comparators should be serviced at intervals no greater than twelve-months and be evidenced by a service report.

9.5 Laboratories holding accreditation for comparative portable hardness testing shall carry out regular in-house checks using suitable reference hardness blocks or samples. These checks shall include as a minimum:

a) a check before use on each day the portable hardness comparator is used;

b) checks when different surface configurations are tested;

c) checks before use whenever the orientation at which the comparator is used is changed (e.g. from vertically upwards to horizontally);

d) checks when a series of tests is to be carried out in the same orientation; these checks should be made at intervals that are sufficiently short to ensure that the portable hardness testing comparator is producing consistent comparative hardness values.

9.6 For portable Brinell, Rockwell, superficial Rockwell or Vickers hardness comparators, reference blocks with a valid certificate of calibration shall be used.

9.7 Test reports issued by laboratories accredited for comparative portable hardness testing shall clearly state that the hardness tests results are comparative.

9.8 There is no general process for accurate conversion of one hardness value to another hardness scale. Such conversions, therefore, should be avoided, unless a reliable basis can be obtained by comparison tests. Hardness readings reported as a conversion to another hardness scale shall be clearly identified as a conversion, and the basis and method of the conversion shall be specified. They shall not be reported as accredited.

10. Bias and uncertainty

10.1 When a conforming hardness machine is directly verified, calibration offsets in Force, displacement, Indenter geometry and in the timing cycle can be identified. These offsets shall be within the limits permitted for a calibration/testing machine by the relevant hardness standard.
Calibration curves of actual vs. nominal values should be used to select the calibrated Force and displacement values to be used in hardness calculations (for indirect validation or subsequent hardness testing). This removes systematic offsets in hardness and simplifies the uncertainty calculations. Uncorrected direct calibration offsets in force and displacement shall be converted to an equivalent offset in Hardness (using the best available sensitivity coefficient) and added to the uncertainty.

Hardness offsets in indenter geometry and timing cycle may be used to correct the measured Hardness values only if the sensitivity coefficient used can be validated for the material being tested and if the associated uncertainty of the correction is included in the uncertainty evaluation. Note: Sensitivity coefficients for deviations in indenter geometry and timing cycle are hard to obtain and harder to validate for a specific material being tested. If no correction for these effects is made, they remain as part of the difference obtained in the indirect verification between the measured value and certified value of a reference block.

The difference between the indirect validation result and the certified value of the reference hardness block includes an aggregate of all uncorrected systematic hardness offset/biases of the machine plus one instance of the statistical variation of the measure value. The latter component is not a systematic offset but is a random contribution due to the small number of tests performed in an indirect validation, the repeatability of the machine, and the uncertainty of the certified value of the hardness reference block. The combination of the machine uncertainty and the reference block uncertainty is an estimate of the uncertainty of the measured value of bias. In many cases the uncertainty in a measured offset/bias is larger than the offset/bias value itself. Therefore, the offset in hardness determined in an indirect verification should not be applied as a correction to measured hardness values unless it exceeds the 95% confidence limits of the combined uncertainty of the machine being verified and the reference hardness block uncertainty. If the offset in hardness determined in an indirect verification is not used as a correction, it shall be treated as an uncertainty component in the indirect verification and future hardness results. If a correction is made to the values of hardness used to make and indirect verification, that indirect verification result is only valid if the same correction method is applied to all future hardness measurements obtained by that combination of machine and indenter.

Note: Increasing the number of hardness measurements included to determine the average value of a reference hardness block will reduce the relative size of the random contribution to the uncertainty in the average measured hardness and therefore to the calculated hardness bias. This can be achieved by taking more measurements in an indirect verification or by averaging the results of a number of indirect verifications performed by the same hardness machine using the same indenter on the same reference hardness block.

Note: Deviations in indenter geometry cause both an error in the calculation of contact area and a material dependent change in the indentation response due to a change in the applied strain of an indentation at a particular stress (applied force). The latter causes a real change in indent size that is not corrected by the application of the geometrical area of contact correction. Therefore, sensitivity coefficients based only upon a correction in effective Area of contact, do not take into account the effect of difference in applied strain. Sensitivity coefficients for deviations in indenter geometry based upon an empirical determination include both effects, but are only valid for the specific material used to determine them.

Deviations in indentation timing cycle from the target values given in the relevant standard cause a change in measured hardness that depends upon the properties of the material being tested (e.g. due to the indentation creep rate, which depends strongly upon both material composition and microstructure). The sensitivity of hardness to timing cycle is, therefore, very material dependent and sensitivity coefficients are only valid if determined upon the material being tested.

10.2 Direct and indirect verification certificates for hardness machines shall contain an estimate of the uncertainty of measurement for each measurement made. Hardness testing laboratories should also report an estimate of the uncertainty of measurement for hardness values obtained during a test.

10.3 The estimate for the uncertainty of measurement should include all significant contributions of uncertainty and be calculated in accordance with the GUM as explained in the UKAS document M3003 *“The Expression of Uncertainty and Confidence in Measurement*.” It may also be calculated using the informative annex given in the relative hardness standard or calculated in accordance with the Euramet document cg-16 version 2.0 “Guidelines on the Estimation of Uncertainty in Hardness Measurements” (Previously EA-10/16). It is to be expected that using different procedures for estimating and evaluating uncertainty will generate different uncertainty values. Only uncertainty values obtained using the same method may be directly compared.

10.4 Indenter certificates shall contain sufficient geometric information to calculate the estimated uncertainty of measurement and bias values for the hardness machine in which it is to be used.

11. Bibliography

11.1 International Standards Organisation (ISO), *Guide to the Expression of Uncertainty in Measurement (GUM)*

11.2 European Association of National Metrology Institutes (EURAMET) cg-16, *Guidelines on the Estimation of Uncertainty in Hardness Measurements*

11.3 United Kingdom Accreditation Service (UKAS), M3003, *The Expression of Uncertainty and Confidence in Measurement*

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12. Glossary of terms

Bias	A (systematic) difference between the true value and the measured or calculated value.
Calibration	Comparison of measurements performed by a measuring instrument to establish the relationship between the indicated values and known values of a measured quantity. *Note: The term 'calibration' as defined internationally does not include adjustment of the instrument.*
Direct verification	Verification by direct measurement of the applied force, the displacement measuring system, the indentation time cycle, and the indenter geometry.
Indirect verification	The verification of a machine using *reference hardness blocks*.
Repeatability	A measure of a hardness machine's ability to obtain the same result when repeated measurements are made under the same conditions.
Resolution	The value of the smallest scale or digital interval displayed by the machine.
Scale	A hardness scale as categorized by hardness standards according to indenter type and force applied.
Primary standardising machine	A national hardness machine which is used to calibrate *primary reference hardness blocks* and is directly traceable to the S.I.
Calibration machine	(Also known as "Standardizing machine" in ASTM hardness standards). A hardness machine which is used to calibrate *reference hardness blocks* and indenters and is directly and indirectly verified annually.
Testing machine	A hardness machine which is used to carry out hardness testing and is directly verified on installation and indirectly verified annually.
Primary reference block	A reference block with a specific hardness value calibrated using a *primary standardising machine*.
Reference block	A reference block with a specific hardness value calibrated using a *calibration machine*. (Also known as "Test Block" in ASTM hardness standards).
Uncertainty	Parameter representing the range of values that could be attributed to the measurand, at a specified confidence level, given the result of a measurement.

13. Reference standards

- ISO 4545-1:2023 Metallic materials. Knoop hardness test. Test method
- ISO 4545-2:2017 Metallic materials. Knoop hardness test. Verification and calibration of testing machines
- ISO 4545-3:2017 Metallic materials. Knoop hardness test. Calibration of reference blocks
- ISO 4545-4:2018 Metallic materials. Knoop hardness test. Table of hardness values

- ISO 6506-1:2014 Metallic materials. Brinell hardness test. Test method
- ISO 6506-2:2018 Metallic materials. Brinell hardness test. Verification and calibration of testing machines
- ISO 6506-3:2014 Metallic materials. Brinell hardness test. Calibration of reference blocks
- ISO 6506-4:2014 Brinell hardness test. Table of hardness values

- ISO 6507-1:2023 Metallic materials. Vickers hardness test. Test method
- ISO 6507-2:2018 Metallic materials. Vickers hardness test. Verification and calibration of testing machines
- ISO 6507-3:2018 Metallic materials. Vickers hardness test. Calibration of reference blocks
- ISO 6507-4:2014 Vickers hardness test. Table of hardness values

- ISO 6508-1:2023 Metallic materials. Rockwell hardness test. Test method
- ISO 6508-2:2023 Metallic materials. Rockwell hardness test. Verification and calibration of testing machines and indenters
- ISO 6508-3:2023 Metallic materials. Rockwell hardness test. Calibration of reference blocks

- ASTM E10-23 Standard Test Method for Brinell Hardness of Metallic Materials
- ASTM E18-24 Standard Test Methods for Rockwell Hardness of Metallic Materials
- ASTM E92-23 Standard Test Methods for Vickers Hardness and Knoop Hardness of Metallic Materials
- ASTM E384-22 Standard Test Method for Microindentation Hardness of Materials