

# **Guidelines on the Estimation of Uncertainty in Hardness Measurements**

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# Calibration Guide

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### **Purpose**

This document provides guidance for calibration and testing laboratories involved in hardness measurements, as well as their assessors. It has been produced to improve harmonization in determination of uncertainties in hardness measurements with special emphasis on Rockwell hardness. It is based on international standards with respect to traceability requirements and calibration procedures.

### **Authorship**

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### Contents

1	INTRODUCTION .....	1
2	PARAMETERS THAT AFFECT THE UNCERTAINTY OF INDENTATION HARDNESS MEASUREMENT .....	4
	2.1 Reference/test material	4
	2.2 Hardness machine	6
	2.3 Environment	6
	2.4 Operator	7
3	GENERAL PROCEDURE FOR CALCULATING THE UNCERTAINTY OF HARDNESS MEASUREMENT .....	7
4	APPLICATION TO THE ROCKWELL C SCALE: EVALUATION AND PROPAGATION OF UNCERTAINTY.....	9
	4.1 Calibration uncertainty of hardness testing machines (direct calibration method)	9
	4.2 Calibration uncertainty of the indirect calibration method	13
5	REFERENCES.....	19

## Guidelines on the Estimation of Uncertainty in Hardness Measurements

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### 1 INTRODUCTION

- 1.1 In the field of hardness measurement a wide variety of methods and equipment is applied which may differ according to the material. A hardness measurement is useful when the — results obtained at different sites are compatible to within a determined interval of measurement uncertainty. The guide aims to demonstrate the concepts of measurement uncertainty applied to this special field. Only uncertainties of the commonly used indentation hardness measuring methods for metals (Brinell, Rockwell, Vickers) are discussed, for the ranges generally employed in engineering practice where universal metrological methods have already been implemented in industrial countries.
- 1.2 A hardness value is the result of a measurement performed on a test piece under standard conditions, and it is based on an agreed convention. The hardness determination is essentially performed in two steps:
1. An indentation is made under prescribed conditions,
  2. The determination of a characteristic dimension of the indentation (mean diameter, mean diagonal or indentation depth).
- 1.3 The dissemination of hardness scales is based on three main elements:
- a) **the hardness scale definition:** description of the measurement method, the relevant tolerances of the quantities involved and the limiting ambient conditions.
  - b) **the hardness reference machine:** metrological devices that materialise the hardness scale definitions. Distinction should be made between *primary standard machines*, which constitute the best possible realisation of the hardness scale definitions, and *calibration machines*, used for the industrial production of hardness reference blocks.
  - c) **the hardness reference block:** One may distinguish between *primary hardness reference blocks*, calibrated by primary hardness standard machines and used when the highest accuracy is required, e.g. for verification and calibration of hardness calibration machines, and *hardness reference blocks* intended mainly for the verification and calibration of industrial hardness testing machines.
- 1.4 Figure 1.1 shows the four-level structure of the metrological chain necessary to define and disseminate hardness scales. Note that at each level both direct calibration and indirect calibration are required. Direct calibration gives any possible reference to mass, length and

time national standards, and checks the conformity to tolerances required by the scale definition. Indirect calibration is required because a number of factors, not yet completely defined (e.g. displacement-time pattern during the indentation, shape irregularities and mechanical performances of the indenter) cannot be evaluated by direct measurement. Comparisons like international comparisons for the Primary Hardness Standard Machines, comparisons with Primary Hardness Standard Blocks for the Hardness Calibration Machines and finally comparisons with Hardness Reference Blocks for Hardness Testing Machines are considered, therefore, as indirect measurements. Direct calibration and indirect calibration cover, as shown before, different contributions to the uncertainty, so that different expressions of the uncertainty, with different meaning, can be obtained:

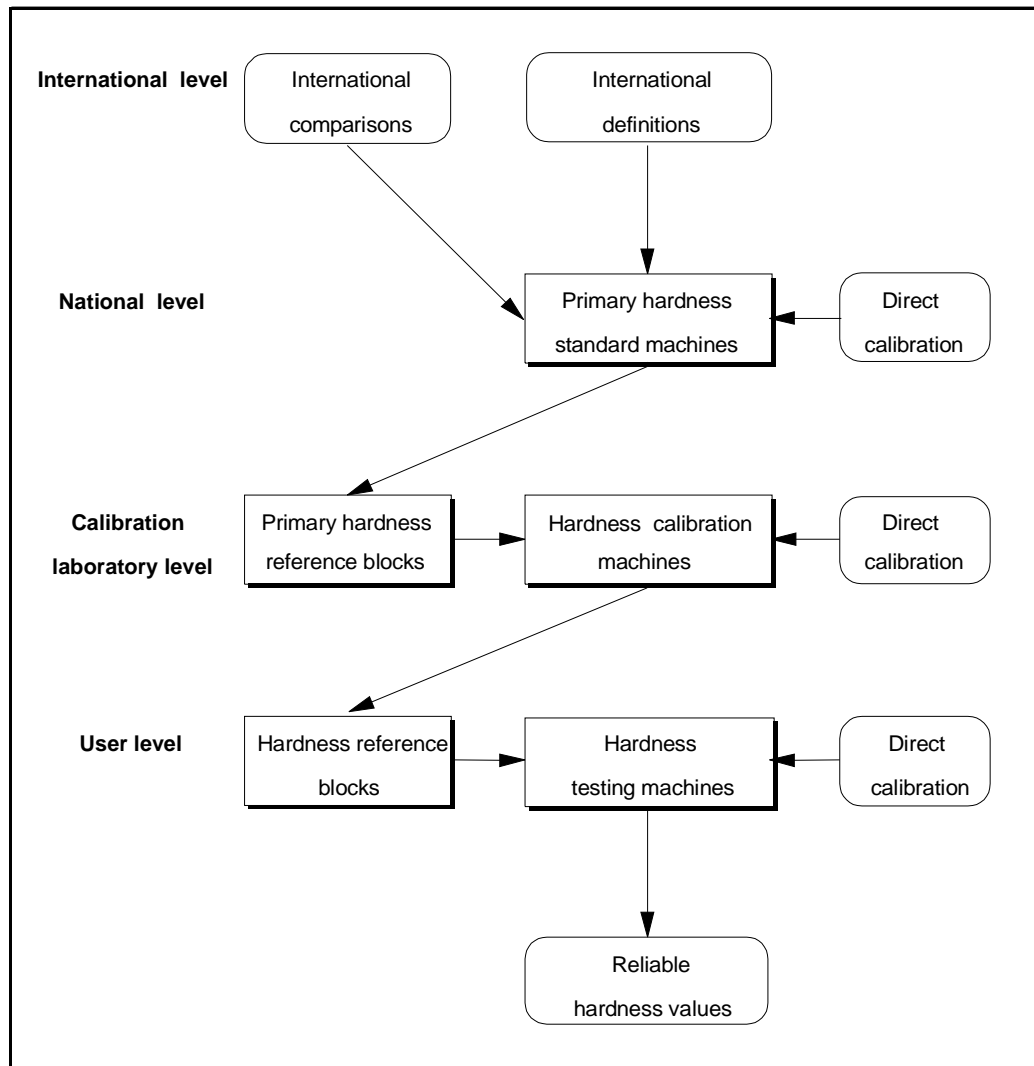
- a) uncertainty of the scale definition, produced by the tolerances adopted and by the lack of definition of some influence factors;
- b) uncertainty of the nominal materialisation of the scale definition, produced by the uncertainty of the factors defined by the scale definitions (taken into account by the direct calibration);
- c) uncertainty of the effective materialisation of the scale definition, produced by the factors not defined by the scale definitions (taken into account by the indirect calibration).

Notice that contribution a) is inherent to the definition itself and therefore shall always be combined with contributions b) and c) that are, at least partially, overlapping, so that one can take the maximum value of the two separate evaluations.

- 1.5 The metrological chain starts at the international level using international definitions of the various hardness scales to carry out international intercomparisons.
- 1.6 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.
- 1.7 No international standards are available for this first step in the materialisation of hardness scales. Due to the small number of laboratories at the national level, their work is regulated by internal operation procedures for the primary machines only and, of course, by the regulations for *international intercomparisons*.
- 1.8 At the **calibration laboratory level**, the *primary hardness reference blocks* are used to qualify the hardness calibration machines, which also have to be calibrated directly and indirectly. These machines are then used to calibrate the *hardness reference blocks* for the user level.
- 1.9 At the **user level**, hardness reference blocks are used to calibrate the industrial hardness testing machines in an indirect way, after they have been directly calibrated.
- 1.10 The stability of hardness scales is essentially underpinned by this two-step calibration procedure for hardness machines:
  - I) *Direct calibration* ensures that the machine is functioning correctly in accordance with the hardness definitions and regarding the appropriate parameters;
  - II) *Indirect calibration* with hardness reference blocks covers the performance of the machine as a whole.
- 1.11 The main requirements for the hardness of reference blocks are stability with time and uniformity over the block surface.

1.12 In some cases hardness blocks calibrated by primary standard machines are used directly for the verification and calibration of industrial hardness testing machines. This is not in line with the four-level structure of figure 1.1, but there are good reasons for it. In hardness metrology the classical rule of thumb - namely that the reference instrument should be an order of magnitude or at least a factor of three better than the controlled device - in many cases cannot be applied.

The uncertainty gap between the national level and the user level is fairly small and each step from one level to the next adds an additional contribution to the total uncertainty; so the four-level hierarchy may lead to uncertainties too large for reliable hardness values at the user level. Most metrological problems of hardness comparison, of error propagation and traceability to standards have their origins in this fact. The calculations in section 4 illustrate this problem.



**Fig. 1.1: The structure of the metrological chain for the definition and dissemination of hardness scales**

## 2 PARAMETERS THAT AFFECT THE UNCERTAINTY OF INDENTATION HARDNESS MEASUREMENT

- 2.0.1 Indentation hardness measurement can often be rightly considered non-destructive since the tested part is still usable afterwards. However, destruction at the actual point of test makes it impossible to verify the uncertainty of the process by a repeated measurement at that same point. It is therefore important that every single measurement be performed to a high degree of accuracy (see section 2.4).
- 2.0.2 There are several influencing parameters that affect the uncertainty of hardness measurements more or less seriously; they are listed in table 2.1 and divided into groups according to their origins:
1. Test piece
  2. Hardness testing machine
  3. Environment
  4. Operator
- 2.0.3 The table lists more than 20 sources of uncertainty which may all contribute significantly to the total uncertainty of a hardness measurement. These sources of uncertainty may not always contribute to every measurement at every level of the metrological chain illustrated in figure 1.1.

### 2.1 Reference/test material

- 2.1.1 Table 2.1 shows that the test piece material introduces a significant number of uncertainties. For example, the *test piece thickness* may affect the measured hardness if the wrong method is selected. The deeper the indent, the thicker the test piece needs to be. Material which is too thin will yield harder results because of the anvilling effect. In addition, if the material is too thin to support the test force during measurement, the indenter itself could be damaged and this will undermine the reliability of any further measurement performed with that indenter.
- 2.1.2 The *surface quality of the test piece* may also considerably influence the results of hardness measurements. A rougher surface would require a greater force and/or a larger indenter to produce a larger indentation. The Brinell method may be the most appropriate since it is less affected by a rough surface than Rockwell or Vickers. Although Brinell measurements are more tolerant of varying finish, there are limits to the permissible surface roughness for this method too. In general, uniformity of surface finish is important for accurate and reproducible results.
- 2.1.3 *Surface cleanliness* is also critical for precise and reproducible hardness measurement. Surface soiling with grease, oxides or dust may cause considerable deviations in the results; moreover, the test material or reference block may even be irreversibly damaged.



**Table 2.1: Parameters that affect the uncertainty of indentation hardness measurement**

Influencing factor	Source of uncertainty	Remarks	Parameters considered for calculation	
<b>1. Test piece</b>	Test piece thickness too low			
	Stiffness of the support			
	Grain structure too coarse	Only relevant, if the chosen test method is not appropriate.		
	Surface roughness			
	Inhomogeneous distribution of hardness			
	Surface cleanliness			
<b>2. Hardness testing machine</b>				
a) Machine frame	Friction loss			
	Elastic deflection			
	Misalignment of the indenter holder			
b1) Depth measuring system	Indicating error	Only relevant for Rockwell	indentation depth	$h$
	Poor resolution			
	Nonlinearity			
	Hysteresis			
b2) Lateral measuring system	Indicating error	Only relevant for Brinell, Vickers, Knoop		
	Poor resolution			
	Numerical aperture of lens or illuminator			
	Inhomogeneous illumination of the indentation			
c) Force application system	Deviation from nominal forces		preliminary/total test force	$F_0, F$
	Deviation from time intervals of the testing cycle		preliminary/total test force dwell time	$t_0, t$
	Force introduction			
	Overrun of test forces		indentation velocity	$v$
d) Indenters	Deviation from the ideal shape		indenter radius and angle	$r, \alpha$
	Damage			
	Deformation under force			
<b>3. Environment</b>	Temperature deviation or drift			
	Vibration and shocks			
<b>4. Operator</b>	Wrong selection of test method			
	Handling, reading, evaluation errors			

## **2.2 Hardness machine**

- 2.2.1 The design, assembly and condition of the hardness testing machine are all critical to accurate results. Excessive friction can cause bias and non-repeatability. Even instruments that are operated properly can give poor results due to excessive friction in the force applying system. Similar uncertainty contributions due to small amounts of friction can be expected from the depth measuring system.
- 2.2.2 Excessive deflections of the supporting frame of the testing machine and the test piece support system can cause problems too. Deviations of 1 to 3 hardness units are not uncommon due to improper support of the test piece and excessive deflection of the instrument's frame.
- 2.2.3 Due to the very small dimensions that are measured, the measuring system is critical. For example, one regular Rockwell scale unit is equivalent to only 2  $\mu\text{m}$  indentation depth and the superficial scale is half of that, so measuring system uncertainty is very important.
- 2.2.4 The force application system must constantly apply accurate forces. High-quality measuring equipment should be able to apply forces well within the limits of  $\pm 1.0\%$  for the user level, and even within 0.1% of the nominal force for calibration machines.
- 2.2.5 Application of the forces requires that both the velocity and the dwell time of the forces be defined. Variations of testing cycle parameters that may occur with some manually controlled machines can produce variations in the result of up to 1 HRC at 60 HRC. Softer materials and materials subject to work hardening could give significantly higher uncertainties. In these cases contributions of dwell time uncertainty and indentation velocity shall be evaluated specifically for the material tested.
- 2.2.6 The properties of the indenter also influence the uncertainty of hardness measurements. It is relatively easy to manufacture a ball to the required shape. However, the ball holder is the main source of uncertainty.
- 2.2.7 Diamond indenters are more difficult to manufacture to the required shape. The potential sources of uncertainty are significant, but in this context it is not necessary to categorise the effect of each in detail. It is important to note here that the best Rockwell diamond indenters manufactured today will exhibit variations up to  $\pm 0.5$  HRC when compared on the same testing machine. Lower quality indenters will give significantly larger variations.

## **2.3 Environment**

- 2.3.1 Ambient *temperature* may have considerable influence on the results of hardness measurements, especially if small lengths have to be determined. The lower limit for Vickers indentations is 20  $\mu\text{m}$ , and the minimum depth for Rockwell scales N and T is only 6  $\mu\text{m}$  to 7  $\mu\text{m}$ . According to the relevant standards, the temperature ranges are 10°C to 35°C for the test methods and (23 $\pm$ 5)°C for the calibration of reference blocks. These ranges are too wide for some hardness scales, but operation outside these ranges should in any case be cause of concern. If this is unavoidable, comparative measurements should be performed to assess the influence of temperature.
- 2.3.2 *Vibrations, electrical interference and lack of cleanliness*, can cause significant problems that are difficult to quantify. Ultra-low force microhardness measurements of course require an absolutely vibration-free environment, whereas vibration requirements for test forces above 200 mN are not so critical.

## 2.4 Operator

Measurement positions on the surface of the sample become important in many cases. Measurements, for instance, near the edge of a piece or at points close to each other must be properly located to ensure accurate results. Uncertainties of up to 2 HRC are not uncommon here. Overall monitoring of the operation is very important. Some modern testing machines have features that minimise operator influence; nevertheless, the latter is still essential for a successful hardness measurement.

## 3 GENERAL PROCEDURE FOR CALCULATING THE UNCERTAINTY OF HARDNESS MEASUREMENT

The following procedure is based on EA/4-02 [1] (cf. worked examples in section 4).

- a) Express the relationship between the measured hardness  $H$  (output quantity) and the input quantities  $X_i$  (model function) in mathematical terms:

$$H = f(X_1, X_2, \dots, X_N) \quad (1)$$

Notice that in the case of Hardness a mathematical relationship connecting input quantities  $X_i$  with the output quantity  $H$  is *not known at the state of the art*. The connection is given by the scale definitions that are empirical procedures. The model function, therefore, does not give much more than a list of factors affecting the measurement results. In practice this is sufficient for establishing a procedure based on EA/4-02, providing that special care is adopted for evaluating standard uncertainties of the input quantities and sensitivity coefficients, as shown here after.

- b) Identify and apply all significant corrections.  
 c) List all sources of uncertainty in the form of an uncertainty analysis in accordance with the following table:

**Table 3.1: Schematic of an ordered arrangement of the quantities, estimates, standard uncertainties, sensitivity coefficients and uncertainty contributions used in the uncertainty analysis of a hardness measurement**

quantity $X_i$	estimate $x_i$	standard uncertainty $u(x_i)$	sensitivity coefficient $c_i$	contribution to the standard uncertainty $u_i(H)$
$X_1$	$x_1$	$u(x_1)$	$c_1$	$u_1(H)$
...	...	...	...	...
$X_n$	$x_n$	$u(x_n)$	$c_n$	$u_n(H)$
Hardness	$H$			$u(H)$

The quantities in table 3.1 are defined as follows:

- $X_i$  quantities, reported in table 2.1, affecting the measurement result  $H$ . As said in 1.4 the uncertainty can be evaluated in two separate ways: the first way involving the physical quantities used for the scale definitions (forces, lengths, times, velocities etc.), refers to the direct calibration; the second way, involving all the factors of influence present in practice, refers to the indirect calibration. Notice that one could suppose that this second way contains all the uncertainty contributions, therefore can alone give the uncertainty value

required, but this is not always true. For instance it is possible to perform a very careful indirect calibration that produces an uncertainty lower than the uncertainty produced by the tolerances accepted for direct calibration [2]. For this reason both ways shall be followed and the larger of the two uncertainty values obtained taken as the result.

$x_i$  estimate values of the quantities  $X_i$

$u(x_i)$  standard uncertainties of the estimates  $x_i$ . Some ways can be followed for determining  $u(x_i)$ . For the part connected with the uncertainty of hardness scale definitions one shall take the tolerance fields of the definition [3] as variability fields, and evaluate the uncertainty contributions of type B. Type B uncertainties shall be used in any case when only a declaration of conformity is available. For the part connected with direct calibration it is possible to determine  $u(x_i)$  by the uncertainty declared in calibration certificates of the measurement instruments used for direct measurements. For the part connected with indirect calibration, that is comparisons performed using hardness blocks, the relevant uncertainty of type A shall be evaluated.

$c_i$  is the sensitivity coefficient associated with the input estimate  $x_i$ . The sensitivity coefficient  $c_i$  describes the extent to which the hardness  $H$  is influenced by variations of the input estimate  $x_i$ . As said before at the state of the art the mathematical connection between  $x_i$  and  $H$  is unknown, therefore the sensitivity coefficients shall be evaluated experimentally by the change  $\Delta H$  in the hardness  $H$  due to a change  $\Delta x_i$  in the input estimate  $x_i$  as follows:

$$c_i \approx \left. \frac{\Delta H}{\Delta x_i} \right|_{x_1=x_1, \dots, x_n=x_n} \quad (2)$$

The experimental evaluation of the sensitivity coefficients is usually time consuming, therefore usually it is advantageous to use the experimental results given in literature [4, 5] and adopted for the examples attached, but one shall be careful when the relevant factors depend on the characteristics of the material tested (dwell time and indentation velocity). In this case some experiments with the specific material are necessary.

$u_i(H)$  is the contribution to the standard uncertainty associated with the hardness  $H$  resulting from the standard uncertainty  $u(x_i)$  associated with the input estimate  $x_i$ :

$$u_i(H) = c_i u(x_i) \quad (3)$$

d) For uncorrelated input quantities the square of the standard uncertainty  $u(H)$  associated with the measured hardness  $H$  is given by:

$$u^2(H) = \sum_{i=1}^n u_i^2(H) \quad (4)$$

e) Calculate for each input quantity  $X_i$  the contribution  $u_i(H)$  to the uncertainty associated with the hardness  $H$  resulting from the input estimate  $x_i$  according to Eqs. (2) and (3) and sum their squares as described in Eq. (4) to obtain the square of the standard uncertainty  $u(H)$  of the hardness  $H$ .

f) Calculate the expanded uncertainty  $U$  by multiplying the standard uncertainty  $u(H)$  associated with the hardness  $H$  by a coverage factor  $k=2$ :

$$U = ku(H) \quad (5)$$

Should the effective degrees of freedom  $\nu_{\text{eff}}$  in exceptional cases be less than 15, then calculate the coverage factor  $k$  according to EA/4-02, Annex E [1].

- g) Report the result of the measurement as follows: in calibration certificates, the complete result of the measurement comprising the estimate  $H$  of the measurand and the associated expanded uncertainty  $U$  shall be given in the form  $(H \pm U)$ . To this an explanatory note must be added which in the general case should have the following content:

The reported expanded uncertainty of measurement has been obtained by multiplying the combined standard uncertainty by the coverage factor  $k=2$  that, for a normal distribution, corresponds to a confidence level  $\rho$  of approximately 95%. The combined standard uncertainty of measurement has been determined in accordance with EA/4-02 [1].

## **4 APPLICATION TO THE ROCKWELL C SCALE: EVALUATION AND PROPAGATION OF UNCERTAINTY**

The relevant standard documents [2] require that both direct and indirect calibration methods be used, at least with new, revised or reinstalled hardness testing machines. It is always good practice to use both calibration methods together.

### ***4.1 Calibration uncertainty of hardness testing machines (direct calibration method)***

- 4.1.1 The direct calibration method is based on the direct measurement of the hardness scale parameters prescribed by ISO 6508-2 [2]. Even though it is not possible to establish an analytical function to describe the connection between the defining parameters and the hardness result [4], some experiments [5] do allow, as described in section 3, to evaluate uncertainty propagation. Yet one should be careful in the application because some of the parameters are primarily connected with the measuring system (preliminary test force, total test force, indentation depth, indenter geometry, frame stiffness), whereas others refer to the measurand (creep effect, strain-hardening effect).
- 4.1.2 The measurand related parameters can be described as an indication based on results obtained with hardness reference blocks, but should be evaluated directly for the specific measurand. The creep effect depends on both the measuring system and the material characteristics; the amount of creep is a function of the creep characteristic of the material, also depending on the time required by the measuring system to register the force. For a manual zeroing machine, creep has generally stopped when zero is finally reached. Even automatic machines are more or less prompt. A machine that takes 5 s to apply the preliminary test force produces a different creep relaxation than a machine taking only 1 s, and the strict observance of a 4 s force dwell-time will not help to obtain compatible results.
- 4.1.3 There is call for caution in interpreting numerical values because the results obtained with old manual machines cannot represent those of a modern automatic hardness testing machine, designed to produce indentations in the shortest possible time.
- 4.1.4 The evaluation of uncertainty is described in the relevant EA/4-02 document [1]. The uncertainty calculation must be done in different ways, depending on the types of data available. The first step is the evaluation of the appropriate variances corresponding to the measurement parameters involved (independent variables).

4.1.5 The measurement results given in a calibration certificate, with the uncertainty usually quoted for  $k=2$  coverage factor, permit the calculation of the standard uncertainty. It is sufficient to divide the given uncertainty by the stated coverage factor. Conformity declaration can also be used to evaluate the standard uncertainty, taking the tolerance interval  $\pm a$  into account. A rectangular distribution function should be used, with equivalent variance  $u^2 = a^2/3$ .

4.1.6 The second step is the calculation of the combined standard uncertainty. Theoretically, if the hardness  $H$  is the measurand (dependent variable), it can be represented as a function of the measurement independent variables. The symbols used are indicated in table 4.1:

$$H = f(F_0; F; r; \alpha; t_0; t; v; h; N; S) \quad (6)$$

More explicitly, the equation is:

$$H = N - \frac{h}{S} + \sum \left( \frac{\partial H}{\partial x_i} \right) \Delta x_i \quad (7)$$

where  $x_i$  are the independent variables in eq. (9).

4.1.7 Using the appropriate sensitivity coefficients, namely the partial derivatives of the dependent variable  $H$  against the independent variables  $x_i$ , one obtains the formula for evaluating the uncertainty propagation in the approximation of uncorrelated independent variables:

$$u^2(H) \approx \sum_{i=1}^n u_i^2(H) = \sum_{i=1}^n c_i^2 u^2(x_i) \quad (8)$$

In practice, the partial derivatives can be approximated by the incremental ratios:

$$u^2(H) = \left( \frac{\Delta H}{\Delta F_0} \right)^2 u^2(F_0) + \left( \frac{\Delta H}{\Delta F} \right)^2 u^2(F) + \left( \frac{\Delta H}{\Delta r} \right)^2 u^2(r) + \left( \frac{\Delta H}{\Delta \alpha} \right)^2 u^2(\alpha) + \left( \frac{\Delta H}{\Delta t_0} \right)^2 u^2(t_0) + \left( \frac{\Delta H}{\Delta t} \right)^2 u^2(t) + \left( \frac{\Delta H}{\Delta v} \right)^2 u^2(v) + \left( \frac{\Delta H}{\Delta h} \right)^2 u^2(h) \quad (9)$$

4.1.8 The standard uncertainty can be evaluated for different conditions. As an example, Table 4.2 shows the evaluation of the standard uncertainty  $u(H)$ , and the expanded uncertainty with coverage factor  $k=2$ , for a conformity assessment of hardness testing machines and indenters to the relevant standard [2]. This was done using the appropriate tolerances to calculate type B standard uncertainties.

**Table 4.1: symbols used**

$H$	Measured hardness	$t$	total test force dwell time	$u_d$	uncertainty of hardness scale definition
$F_0$	Preliminary test force	$v$	indentation velocity	$u_m$	uncertainty of primary hardness standard machine
$F$	total test force	$h$	indentation depth	$u_s$	stability uncertainty of calibration machine
$r$	Indenter radius	$N$	constant number dependent by the scale	$u_f$	fitting uncertainty
$\alpha$	indenter angle	$S$	constant number dependent by the scale	$\nu_j$	degrees of freedom
$t_0$	Preliminary test force dwell time				
$H_b$	mean hardness measurement result of primary hardness reference block		$s_c$		Standard deviation of the measurements $H_c$
$H_{bi}$	single hardness measurement result of primary hardness reference block		$s_{ci}$		Standard deviation of the measurements $H_{ci}$
$u_{bd}$	Calibration uncertainty of primary hardness reference blocks considering the scale definition		$H_c$		Mean hardness values of the scale of the calibration machine
$u_{bm}$	Calibration uncertainty of primary hardness reference blocks considering the uncertainty of the primary hardness standard machine		$H_{ci}$		Single hardness values of the scale of the calibration machine
$s_b$	Standard deviation of the measurement $H_b$		$u_{cdf}$		Calibration machine uncertainty considering the scale definition uncertainty and the fitting uncertainty
$s_{bi}$	Standard deviation of the measurements $H_{bi}$		$u_{cmf}$		Calibration machine uncertainty considering the primary standard machine uncertainty and the fitting uncertainty
$u_{cd}$	Calibration uncertainty of the calibration machine considering the scale definition		$u_{cdur}$		Calibration machine uncertainty considering the scale definition uncertainty and the calibration results uncorrected
$u_{cm}$	Calibration uncertainty of the calibration machine considering the uncertainty of the primary hardness standard machine		$u_{cmu}$		Calibration machine uncertainty considering the primary standard machine uncertainty and the calibration results uncorrected
$\Delta H$	Correction value				

**Table 4.2: Evaluation of the uncertainty propagation for conformity assessment of the hardness testing machine and indenter**

$x_i$	$a_i$	$u^2(x_i) = \frac{a_i^2}{3}$	Sensitivity coefficients at different hardness levels $c_i = \frac{\Delta H}{\Delta x_i}$			Contributions to $u^2(H)/HRC^2$ at different hardness levels $u^2(H) \approx \sum_{i=1}^n u_i^2(H) = \sum_{i=1}^n c_i^2 u^2(x_i)$		
			20 to 25	40 to 45	60 to 65	20 to 25	40 to 45	60 to 65
$F_0/N$	2	$1.3 \cdot 10^0$	$1.2 \cdot 10^{-1}$	$7.0 \cdot 10^{-2}$	$5.0 \cdot 10^{-2}$	$1.9 \cdot 10^{-2}$	$6.4 \cdot 10^{-3}$	$3.3 \cdot 10^{-3}$
$F/N$	15	$7.5 \cdot 10^{+1}$	$-4.0 \cdot 10^{-2}$	$-3.0 \cdot 10^{-2}$	$-2.0 \cdot 10^{-2}$	$1.2 \cdot 10^{-1}$	$6.8 \cdot 10^{-2}$	$3.0 \cdot 10^{-2}$
$\alpha/^\circ$	0.35	$4.1 \cdot 10^{-2}$	$1.3 \cdot 10^{+0}$	$8.0 \cdot 10^{-1}$	$4.0 \cdot 10^{-1}$	$6.9 \cdot 10^{-2}$	$2.6 \cdot 10^{-2}$	$6.6 \cdot 10^{-3}$
$r/mm$	0.01	$3.3 \cdot 10^{-5}$	$1.5 \cdot 10^{+1}$	$3.0 \cdot 10^{+1}$	$5.0 \cdot 10^{+1}$	$7.4 \cdot 10^{-3}$	$3.0 \cdot 10^{-2}$	$8.3 \cdot 10^{-2}$
$h/\mu m$	1	$3.3 \cdot 10^{-1}$	$-5.0 \cdot 10^{-1}$	$-5.0 \cdot 10^{-1}$	$-5.0 \cdot 10^{-1}$	$8.3 \cdot 10^{-2}$	$8.3 \cdot 10^{-2}$	$8.3 \cdot 10^{-2}$
$v/(\mu m/s)$	25	$2.1 \cdot 10^{+2}$	$-2.0 \cdot 10^{-2}$	$0.0 \cdot 10^0$	$3.0 \cdot 10^{-2}$	$8.4 \cdot 10^{-2}$	$0.0 \cdot 10^0$	$1.9 \cdot 10^{-1}$
$t_0/s$	1.5	$7.5 \cdot 10^{-1}$	$1.0 \cdot 10^{-2}$	$5.0 \cdot 10^{-3}$	$4.0 \cdot 10^{-3}$	$7.5 \cdot 10^{-5}$	$1.9 \cdot 10^{-5}$	$1.2 \cdot 10^{-5}$
$t/s$	2	$1.3 \cdot 10^0$	$-7.0 \cdot 10^{-2}$	$-4.0 \cdot 10^{-2}$	$-3.0 \cdot 10^{-2}$	$6.4 \cdot 10^{-3}$	$2.1 \cdot 10^{-3}$	$1.2 \cdot 10^{-3}$
TOTAL	$u^2/HRC^2 = \sum u_i^2/HRC^2$					0.39	0.22	0.40
Standard uncertainty	$u/HRC$					0.62	0.46	0.63
Expanded uncertainty	$U/HRC = ku/HRC$					1.25	0.93	1.26

4.1.9 Table 4.3 shows the evaluation of standard and expanded uncertainty for calibration certificates for the hardness testing machine and indenter. Here the example is for the hardness level 20 HRC to 25 HRC. Note that the differences between the parameter and nominal values are known, together with their uncertainties, and it is therefore possible to estimate both a correction  $\Delta H_i$  and its uncertainty  $u(\Delta H_i)$  using the same sensitivity coefficients as before.

4.1.10 Whilst in the case of type B uncertainty contributions the degrees of freedom  $\nu_i$  of the various parameters can be considered large enough to apply the Gaussian distribution, in this case  $\nu_i$  depends on the adopted measurement procedure. Table 4.3 quotes typical values of  $\nu_i$ .



**Table 4.3: Evaluation of the uncertainty propagation in calibration certificates for the hardness testing machine and for the indenter for 20 HRC to 25 HRC hardness level**

$X_i$	Certificate data			$c_i = \frac{\Delta H}{\Delta X_i}$	Measured hardness		
	$\Delta X_i$	$U_i (2\sigma)$	$\nu_i$		$\Delta H_i$	$u_i^2(H)$	$u_i^4(H)/\nu_i$
					HRC	HRC <sup>2</sup>	HRC <sup>4</sup>
$F_0 / N$	0.8	0.2	8	$1.2 \cdot 10^{-1}$	0.10	$1.4 \cdot 10^{-4}$	$2.6 \cdot 10^{-9}$
$F / N$	-4.3	1.5	8	$-4.0 \cdot 10^{-2}$	0.17	$9.0 \cdot 10^{-4}$	$1.0 \cdot 10^{-7}$
$\alpha / ^\circ$	0.2	0.1	8	$1.3 \cdot 10^0$	0.26	$4.2 \cdot 10^{-3}$	$2.2 \cdot 10^{-6}$
$r / mm$	0.007	0.002	8	$1.5 \cdot 10^{+1}$	0.11	$2.3 \cdot 10^{-4}$	$6.3 \cdot 10^{-9}$
$h / \mu m$	-0.5	0.2	3	$-5.0 \cdot 10^{-1}$	0.25	$2.5 \cdot 10^{-3}$	$2.1 \cdot 10^{-6}$
$v / (\mu m/s)$	20	5	2	$-2.0 \cdot 10^{-2}$	-0.40	$2.5 \cdot 10^{-3}$	$3.1 \cdot 10^{-6}$
$t_0 / s$	1	0.5	3	$1.0 \cdot 10^{-2}$	0.01	$6.3 \cdot 10^{-6}$	$1.3 \cdot 10^{-11}$
$t / s$	1	0.5	3	$-7.0 \cdot 10^{-2}$	-0.07	$3.1 \cdot 10^{-4}$	$3.1 \cdot 10^{-8}$
Total					0.42	0.011	$7.6 \cdot 10^{-6}$
Standard uncertainty $u / HRC$						0.10	
Degrees of freedom						15	
Coverage factor $k$ for confidence level $p = 95\%$						2.13	
Expanded uncertainty $U / HRC = ku / HRC$						0.22	
Where $\Delta H_i = c_i \Delta X_i$ and $u_i^2(H) \approx c_i^2 u^2(x)$							

4.1.11 This method can only be used correctly if nominal values are defined for the various parameters. If, as is the case with current standards, there are parameters which are not defined as nominal values with a given tolerance but as uniform probability intervals, then the reference to a "nominal value" is not possible. In consequence, the uncertainty calculated in this way can only be accepted where there is a preliminary agreement on the "nominal values" of the measurement parameters.

## 4.2 Calibration uncertainty of the indirect calibration method

4.2.0.1 The indirect calibration method is based on a metrological chain. A typical sequence is (cf. Figure 1.1):

- a) definition of the hardness scale;
- b) materialisation of the hardness scale definition by a primary hardness standard machine;
- c) calibration of primary hardness reference blocks for the dissemination of the hardness scale;
- d) calibration of a hardness calibration machine for the industrial production of hardness reference blocks;
- e) calibration of hardness reference blocks;
- f) calibration of industrial hardness testing machines using hardness reference blocks.
- g) hardness measurement performed with industrial hardness testing machines.

4.2.0.2 It is also possible to go directly from step c) to step f), or, after step e) to add the calibration of a frontline hardness testing machine from the industrial quality system and, within the quality system, to calibrate the hardness reference blocks necessary for the calibration of other hardness testing machines used within the quality system itself. Note that after step d) the subsequent steps are repetitions of the previous ones. In consequence, the description of the uncertainty evaluation can be restricted to the first four steps.

#### 4.2.1 Uncertainty $u_d$ of the Rockwell hardness scale definition

4.2.1.1 The evaluation of the uncertainty  $u_d$  of the hardness scale definition and its materialisation is similar to the evaluation of the uncertainty due to the direct calibration method, taking the tolerances prescribed by ISO 6508-3 [3] into account. Table 4.4 presents an example of uncertainty evaluation. Note that uncertainty contributions are of type B, therefore a coverage factor  $k=2$  is used.

**Table 4.4 : Evaluation of the uncertainty  $u_d$  due to the definition of the Rockwell C Scale and its materialisation**

$X_i$	$a_i$	$u^2(x_i) = \frac{a_i^2}{3}$	Sensitivity coefficients at different hardness levels			Contributions to $u^2(H)/HRC^2$ at different hardness levels		
			$c_i = \frac{\Delta H}{\Delta X_i}$			$u^2(H) \approx \sum_{i=1}^n u_i^2(H) = \sum_{i=1}^n c_i^2 u^2(x_i)$		
			20 to 25	40 to 45	60 to 65	20 to 25	40 to 45	60 to 65
$F_0/N$	0.2	$1.3 \cdot 10^{-2}$	$1.2 \cdot 10^{-1}$	$7.0 \cdot 10^{-2}$	$5.0 \cdot 10^{-2}$	$1.9 \cdot 10^{-4}$	$6.4 \cdot 10^{-5}$	$3.3 \cdot 10^{-5}$
$F/N$	1.5	$7.5 \cdot 10^{-1}$	$-4.0 \cdot 10^{-2}$	$-3.0 \cdot 10^{-2}$	$-2.0 \cdot 10^{-2}$	$1.2 \cdot 10^{-3}$	$6.8 \cdot 10^{-4}$	$3.0 \cdot 10^{-4}$
$\alpha/^\circ$	0.1	$3.3 \cdot 10^{-3}$	$1.3 \cdot 10^0$	$8.0 \cdot 10^{-1}$	$4.0 \cdot 10^{-1}$	$5.6 \cdot 10^{-3}$	$2.1 \cdot 10^{-3}$	$5.3 \cdot 10^{-4}$
$r/mm$	0.005	$8.3 \cdot 10^{-6}$	$1.5 \cdot 10^{+1}$	$3.0 \cdot 10^{+1}$	$5.0 \cdot 10^{+1}$	$1.9 \cdot 10^{-3}$	$7.5 \cdot 10^{-3}$	$2.1 \cdot 10^{-2}$
$h/\mu m$	0.2	$1.3 \cdot 10^{-2}$	$-5.0 \cdot 10^{-1}$	$-5.0 \cdot 10^{-1}$	$-5.0 \cdot 10^{-1}$	$3.3 \cdot 10^{-3}$	$3.3 \cdot 10^{-3}$	$3.3 \cdot 10^{-3}$
$v/(\mu m/s)$	10	$3.3 \cdot 10^{+1}$	$-2.0 \cdot 10^{-2}$	$0.0 \cdot 10^0$	$3.0 \cdot 10^{-2}$	$1.3 \cdot 10^{-2}$	$0.0 \cdot 10^0$	$3.0 \cdot 10^{-2}$
$t_0/s$	1.5	$7.5 \cdot 10^{-1}$	$1.0 \cdot 10^{-2}$	$5.0 \cdot 10^{-3}$	$4.0 \cdot 10^{-3}$	$7.5 \cdot 10^{-5}$	$1.9 \cdot 10^{-5}$	$1.2 \cdot 10^{-5}$
$t/s$	2	$1.3 \cdot 10^0$	$-7.0 \cdot 10^{-2}$	$-4.0 \cdot 10^{-2}$	$-3.0 \cdot 10^{-2}$	$6.4 \cdot 10^{-3}$	$2.1 \cdot 10^{-3}$	$1.2 \cdot 10^{-3}$
TOTAL $u_d^2/HRC^2 = \sum u_i^2/HRC^2$						0.03	0.02	0.06
Standard uncertainty $u_d/HRC$						0.18	0.13	0.24
Expanded uncertainty $U/HRC = ku_d/HRC$						0.36	0.26	0.47

4.2.1.2 The evaluated values are confirmed by results obtained during international comparisons, in particular that involving the largest number of participants, which shows a spread of results of about  $\pm 0.5$  HRC.

#### 4.2.2 Uncertainty of the materialisation of the Rockwell hardness scale definition

4.2.2.1 To demonstrate an uncertainty evaluation for state of the art characteristics of primary hardness standard machines, one may do a calculation similar to that in table 4.3, taking relevant uncertainties as shown in table 4.5 into account. The results are optimistic because significant parameters, such as the performance of the indenter, are not accounted for, yet these must be considered as inherent in the uncertainty due to the definition. It can be seen that the uncertainty of the machine is almost negligible compared

to the effect of the tolerances given by the definition, with the uncertainty contributions from influencing quantities missing in the definition itself.

**Table 4.5: Evaluation of the uncertainty  $u_m$  based on the state of the art of primary hardness standard machines for the 20 HRC to 25 HRC hardness level.**

$X_i$	Certificate data			$c_i = \frac{\Delta H}{\Delta X_i}$	Measured hardness		
	$\Delta x_i$	$U_i (2\sigma)$	$\nu_i$		$\Delta H_i$	$u_i^2(H)$	$u_i^A(H)/\nu_i$
					HRC	HRC <sup>2</sup>	HRC <sup>4</sup>
$F_0/N$	0.01	0.01	20	$1.2 \cdot 10^{-1}$	$1.2 \cdot 10^{-3}$	$3.6 \cdot 10^{-7}$	$6.5 \cdot 10^{-15}$
$F/N$	0.15	0.05	20	$-4.0 \cdot 10^{-2}$	$-6.0 \cdot 10^{-3}$	$1.0 \cdot 10^{-6}$	$5.0 \cdot 10^{-14}$
$\alpha/^\circ$	0.05	0.02	20	$1.3 \cdot 10^0$	$6.5 \cdot 10^{-2}$	$1.7 \cdot 10^{-4}$	$1.4 \cdot 10^{-9}$
$r/\text{mm}$	0.003	0.001	20	$1.5 \cdot 10^{+1}$	$4.5 \cdot 10^{-2}$	$5.6 \cdot 10^{-5}$	$1.6 \cdot 10^{-10}$
$h/\mu\text{m}$	0.1	0.05	20	$-5.0 \cdot 10^{-1}$	$-5.0 \cdot 10^{-2}$	$1.6 \cdot 10^{-4}$	$1.2 \cdot 10^{-9}$
$\nu/(\mu\text{m/s})$	5	2	10	$-2.0 \cdot 10^{-2}$	$-1.0 \cdot 10^{-1}$	$4.0 \cdot 10^{-4}$	$1.6 \cdot 10^{-8}$
$t_0/s$	0.5	0.2	10	$1.0 \cdot 10^{-2}$	$5.0 \cdot 10^{-3}$	$1.0 \cdot 10^{-6}$	$1.0 \cdot 10^{-13}$
$t/s$	0.5	0.2	10	$-7.0 \cdot 10^{-2}$	$-3.5 \cdot 10^{-2}$	$4.9 \cdot 10^{-5}$	$2.4 \cdot 10^{-10}$
Total					-0.07	0.001	$1.9 \cdot 10^{-8}$
Standard uncertainty $u_m/\text{HRC}$						0.03	
Degrees of freedom						36	
Coverage factor $k$ for confidence level $p = 95\%$						2.03	
Expanded uncertainty $U/\text{HRC} = ku/\text{HRC}$						0.06	
Where $\Delta H_i = c_i \Delta X_i$ and $u_i^2(H) \approx c_i^2 u^2(x)$							

4.2.2.2 The value of the uncertainty is therefore primarily the result of tolerances of the measuring parameters prescribed by relevant standards. Although table 4.4 does not take the contribution due to the primary hardness standard machine into account for the materialisation of the definition itself, it can still be considered a comprehensive evaluation.

#### 4.2.3 Uncertainty of the calibration of Rockwell primary hardness reference blocks

4.2.3.1 The primary hardness reference block is calibrated by a primary hardness standard machine making five hardness measurements  $H_{bi}$ . The mean value  $H_b$  is taken as the hardness value of the block.

4.2.3.2 Repeating the measurement reveals the effects of non-uniformity of the reference block surface and the repeatability of the primary hardness standard machine, including its resolution. Other effects, such as the hardness stability of reference blocks, must be estimated from experience with the reference blocks and their maintenance conditions.

4.2.3.3 Except for a possible drift that must be evaluated separately, the uncertainty  $u_{bd}$  or  $u_{bm}$  of  $H_b$  can be evaluated from the uncertainty due to the scale definition  $u_{ch}$  given in Table 4.4, combined with the standard deviation  $s_b$  of  $H_b$  evaluated using the standard deviation  $s_{bi}$  of the measurements  $H_{bi}$ .

4.2.3.4 The uncertainties  $u_{bd}$  or  $u_{bm}$  are given by:

$$s_{bi} = \sqrt{\frac{\sum_{i=1}^5 (H_{bi} - H_b)^2}{4}} \quad (10)$$

$$s_b = \frac{s_{bi}}{\sqrt{5}} \quad (11)$$

$$u_{bd} = \sqrt{u_d^2 + s_b^2} \quad \text{or} \quad u_{bm} = \sqrt{u_m^2 + s_b^2} \quad (12)$$

4.2.3.5 The calibration certificate shall at least state the value of the standard uncertainty  $u_{bd}$ . Also required is the value  $u_{bm}$ . Explicit values for the uncertainty contributions [5, 6] can be included for information.

#### 4.2.4 Uncertainty of the calibration of Rockwell calibration machines

4.2.4.1 The hardness reference block is calibrated by a hardness calibration making five hardness measurements  $H_{ci}$ . The mean value  $H_c$  is compared with the block hardness  $H_b$  to calibrate the machine for that scale and that hardness ( $\Delta H = H_c - H_b$ ).

4.2.4.2 Repeating the measurement reveals the effects of non-uniformity of the reference block surface and the repeatability of the hardness calibration machine, including its resolution. Therefore, except for the stability of the calibration machine  $u_s$  that must be evaluated separately because it depends on the working conditions, the uncertainty  $u_{cd}$  or  $u_{cm}$  of  $H_c$  can be evaluated by combining the relevant uncertainty due to the hardness reference block  $u_{bd}$  or  $u_{bm}$  with the standard deviation  $s_c$  of  $H_c$  calculated using the standard deviation  $s_{ci}$  of the measurements  $H_{ci}$ .

4.2.4.3 To minimise the uncertainty, the correction  $\Delta H$  should be applied by the measured hardness. To derive the uncertainty  $u_{cdf}$  or  $u_{cmf}$  at any point of the machine scale one should interpolate the results  $\Delta H$ . The uncertainty due to fitting  $u_f$  depends on the structure and the working characteristics of the calibration machine, and should therefore be determined to characterise the machine itself by a calibration on five hardness levels, comparing the least squares parabola with the parabola passing through the three points at the hardness level chosen for the subsequent periodic checks.

4.2.4.4 For the uncertainties  $u_{cdf}$  or  $u_{cmf}$  we have:

$$s_{ci} = \sqrt{\frac{\sum_{i=1}^5 (H_{ci} - H_c)^2}{4}} \quad (13)$$

$$s_c = \frac{s_{ci}}{\sqrt{5}} \quad (14)$$

$$u_{cd} = \sqrt{u_{bd}^2 + s_c^2} \quad \text{or} \quad u_{cm} = \sqrt{u_{bm}^2 + s_c^2} \quad (15)$$

$$u_{cdf} = \sqrt{u_{cd}^2 + u_f^2} \quad \text{or} \quad u_{cmf} = \sqrt{u_{cm}^2 + u_f^2} \quad (16)$$

if the correction  $\Delta H$  is not applied, the uncertainty  $u_{cdu}$  and  $u_{cmu}$  are calculated using:

$$u_{cdu} = \sqrt{u_{cd}^2 + \Delta H^2} \quad \text{or} \quad u_{cmu} = \sqrt{u_{cm}^2 + \Delta H^2} \quad (17)$$

4.2.4.5 The calibration certificate shall at least state the value of the standard uncertainty  $u_{cdf}$ . Also required is the value of  $u_{cmf}$ . Explicit values of the uncertainty contribution [5, 6] can be included for information.

#### **4.2.5 Uncertainty of the calibration of hardness reference blocks and testing machines**

For the calibration of hardness reference blocks and hardness testing machines the same procedures are used as those described above for calibration of primary hardness reference blocks and hardness calibration machines. The formulae given for those cases shall be used.

#### **4.2.6 Numerical example**

The uncertainty evaluation can be set out as in the following example in Table 4.6.

**Table 4.6 Indirect calibration chain - Uncertainty evaluation**

<b>Hardness level</b>	20 to 25	40 to 45	60 to 65
Definition and standard machine uncertainty ( $u_d$ ) (see Table 4.4)	0.18	0.13	0.24
<b>Primary hardness reference block calibration</b>			
Number of indentations	5	5	5
Non-uniformity of primary hardness reference block and machine reproducibility. Relevant standard deviation ( $s_{bi}$ ) (Eq.10)	0.23	0.17	0.12
Standard deviation of the mean of indentations ( $s_b$ ) (Eq.11)	0.10	0.08	0.05
Uncertainty of the hardness value of reference blocks ( $u_{bd}$ or $u_{bm}$ ) (Eq.12)	0.21	0.15	0.25
<b>Calibration of hardness calibration machine</b>			
Number of indentations	5	5	5
Non-uniformity of primary hardness reference block and machine reproducibility. Relevant standard deviation ( $s_{ci}$ ) (Eq.13)	0.29	0.23	0.17
Standard deviation of the mean of indentations ( $s_c$ ) (Eq.14)	0.13	0.10	0.08
Fitting uncertainty $u_f$	0.09	0.04	0.06
Uncertainty of the hardness scale of the calibration machine ( $u_{cdf}$ or $u_{cmf}$ ) (Eq.15 and Eq.16)	0.26	0.18	0.26
<b>Hardness reference block calibration</b>			
Number of indentations	5	5	5
Non-uniformity of hardness reference block and machine reproducibility. Relevant standard deviation ( $s_{bi}$ ) (Eq. 10)	0.29	0.23	0.17
Standard deviation of the mean of indentations ( $s_b$ ) (Eq.11)	0.13	0.10	0.08
Uncertainty of the hardness value of hardness reference blocks ( $u_{bd}$ or $u_{bm}$ ) (Eq.12)	0.29	0.22	0.27
Effective degrees of freedom $\nu_p$	30	26	42
Coverage factor	2.04	2.06	2.02
Expanded uncertainty $U$	0.59	0.44	0.55

## 5 REFERENCES

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