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Result analysis of EURAMET Vickers comparison between INRiM and UME (EURAMET.M.H-K1.b.c)

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ABSTRACT

A bilateral key comparison between INRiM (National Metrology Institute of Italy) and TUBITAK UME (National Metrology Institute of Turkey) had been decided to be organized in the field of Hardness Metrology to determine the consistency of the national hardness standards in both countries realizing Vickers Hardness measurements in accordance with ISO 6507–1:2018 [1] and ISO 6507–3:2018 [2] standards. Widely used Vickers Hardness scales such as HV1 and HV30 constitute the scope of the comparison which was piloted by INRiM. In this paper the procedure and measurement results of the bilateral EURAMET key comparison between the two laboratories are explained.

1. Introduction

A bilateral key comparison between INRiM (Istituto Nazionale di Ricerca Metrologica) and TUBITAK UME (TÜBİTAK Ulusal Metroloji Enstitüsü) was carried out in the field of Hardness Metrology to determine the consistency of the national hardness standards in both countries realizing Vickers Hardness measurements in accordance with ISO 6507–1:2018 [1] and ISO 6507–3:2018 [2] standards. The most widely used Vickers Hardness scales such as HV1 and HV30 constitute the scope of the comparison which was piloted by INRiM.

The blocks used in the comparison were provided by UME. Each NMI measured three hardness levels for both HV1 and HV30 scales on the same transfer standards using their own indenters. Measurements were carried out first by UME as the provider of the blocks, then by the PL (Pilot Laboratory, INRiM), then again by UME for checking the stability of the transfer standards.

This comparison is linked to the CCM key comparison CCM.H-K1.b.c and the hardness scales and levels are selected accordingly. The CCM.H-K1.b.c was realized during 2001–2003 to investigate the metrological equivalence of the national standards among national metrology institutes (NMIs) within the CCM.

2. Transfer standards used

In the bilateral comparison one set of hardness reference blocks, composed of three blocks for three hardness levels were used for both hardness scales HV1 and HV30. Some information related to the hardness reference blocks used in the comparison is given below in Table 1.

You can see below the pictures of the transfer standards used in the comparison below as Fig. 1.

3. Comparison procedure

It was requested that each participant shall assure that the national standards to be used in the comparison at least were in accordance with the ISO 6507–1:2018 [1] and ISO 6507–3:2018 [2] standards. Under these circumstances the components to be calibrated/verified are as follows;

- Force
- Testing cycle
- Indenter geometrical parameters
- Indentation measurement system

Beside management of calibration of the components given above, the blocks were placed in the laboratory one day before the measurements for temperature equilibrium. The measurement steps realized during the comparison measurement are as follows;

- Before the measurements, it was assured that the standardizing machines were working properly in accordance with their design parameters and relevant ISO Hardness Standards requirements.
- Also the anvil where the blocks are seated on and both surfaces of the reference blocks were clean.
- The relevant scale and related indenter and other requirements were mounted/selected etc.
- The ambient temperature was recorded.
- One set of 5 measurements uniformly distributed on the surface of the block was made.
- The ambient temperature was recorded.

4. Reference values of influence parameters

In the calibration of hardness reference blocks, the calibration laboratories realize this work in accordance with some internationally defined standards or accepted definitions. To perform measurements under the same or very similar conditions by the participants it has been significant to agree on reference values of the influence parameters and testing cycles and realize them as much as possible to have more comparable reference systems. Below are the reference values (see Table 2)

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Table 1
Transfer standards used in the comparison.

No	Hardness Value	Serial Number	Producer
1	200	294–472	YAMAMOTO
2	500	294–481	YAMAMOTO
3	800	224–804	YAMAMOTO

and the measurement cycle (see Fig. 2) used in the HV1 and HV30 measurements.

Unification of testing cycle is as important as unification on other parameters affecting the outcome magnitude hardness due to its being a method based quantity. For instance, speeds of load application and removal, duration of the force kept applied on the indenter for realization of indentation etc, all are affecting the hardness measurements. For these reasons it is very important to unify the cycle through which the measurement is performed. In this comparison the reference values were chosen according to the relevant ISO Hardness Standards [1, 2]. Deviations from these values were taken into the uncertainty calculations.

5. Elaboration of the results

As its being a key comparison, the main aim of this comparison is to link the UME measurement values to the Key Comparison Reference Values (KCRVs) of the CCM KC through the PL (INRiM) as a participant of the CCM.H-K1.b.c, had been realized with the participation of the national metrology institutes (NMIs) within the CCM. The measurement results are used to compute the degree of equivalence of UME with the Key Comparison Reference Values of the CCM.H-K1.b.c by linking the measurement results through the PL. At 840 HV1, where the linking is not possible to calculate, the degree of equivalence of UME and INRiM with the Comparison Reference Value (CRV) is computed. The E_n ratio is computed for all ranges.

In line with this situation, each laboratory recorded the measurement results and uncertainty values in the data sheet provided in the annex of the Technical Protocol for each hardness reference block. The PL was responsible for collecting the measurement data from the participants, compiling, elaboration and preparing the reports. The calculation is shown in following steps and made by making use of the relevant equations.

5.1. Linking measurement results to the CCM.H-K1.b.c key comparison reference values (KCRVs)

The EURAMET.M.H-K1.b.c was organized to link this comparison results to the Key Comparison Reference Values (KCRVs) of the last CCM Key comparison (CCM KC) for Vickers hardness (CCM.H-K1), where HV1 and HV30 measurement results are linked to the CCM.H-K1.b and the CCM.H-K1.c values, respectively, except for the 840 HV1 scale comparison where the PL is not consistent with the KCRV of the CCM KC. For the linked scales, the PL had participated in the CCM KC and the link was made through this laboratory measurement and the relevant uncertainty values. The link calculation is given below.

- Calculation of the Linked Comparison Reference Value ($KCRV_{LINK}$):

The PL (INRiM) calculated the Comparison Reference Values ($KCRV_{LINK}$) for this EURAMET bilateral Key Comparison linked to the CCM KCRV by making use of the PL's Degree of Equivalence (DoE) with the CCM KCRV. The DoE includes the deviations (d_{CCM}) of the PL's measurement values from the CCM KCRV and their associated uncertainties ($U_{d_{CCM}}$). These values are reported in the CCM.H-K1 CCM Key Comparison Report.

Table 2
Ref. Values for vickers hardness (HV1 and HV30).

Symbol	Test parameter	Reference value	Start measurement	Stop measurement
F	Total test force for HV1	9.807 N	-	-
	Total test force for HV30	294.2 N	-	-
α	Angle of the indenter	136°	Tip of the indenter	400 µm
V_{app}	Indenter approach speed	≤ 200 µm·s^{-1}	~0% F	~1% F
T_a	Application time of test force	(7±1) s	~1% F	~99% F
T_d	Duration of the total force	(14±1) s	~99% F	~99% F
T	Temperature of test	23°C	Beginning of the test	End of the test

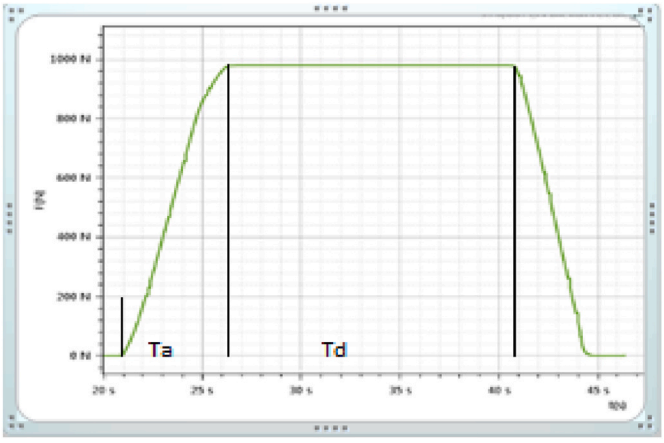

Fig. 2. Identification of HV1 & HV30 testing cycle.

Fig. 1. Transfer standards used in the comparison.

- The PL's CCM KC measurement deviation values (d_{CCM}) provide the link between the results of this comparison and the CCM KCRV and were calculated as shown in Eq [1].

$$d_{CCM} = X_{INRiM_{CCM}} - KCRV_{CCM} \quad (1)$$

where.

$X_{INRiM_{CCM}}$: INRiM's mean measurement value of the CCM KC
$KCRV_{CCM}$: CCM Key Comparison Reference Value

- The linked Key Comparison Reference Values ($KCRV_{LINK}$) for this comparison are calculated from the PL's mean measurement value on the reference block and subtracting the associated d_{CCM} value as shown in Eq [2].

$$KCRV_{LINK} = X_{INRiM} - d_{CCM} \quad (2)$$

where.

X_{INRiM} : INRiM's mean measurement value of this comparison.

- The uncertainty of d_{CCM} (the deviation of the PL from the CCM KCRV) at a 95% level of confidence is calculated as in Eqn [3]:

$$U^2_{d_{CCM}} = U^2_{INRiM_{CCM}} + U^2_{KCRV_{CCM}} \quad (3)$$

where.

$U_{INRiM_{CCM}}$: uncertainty in INRiM's mean measurement value of the CCM KC
$U_{KCRV_{CCM}}$: uncertainty in the associated CCM KCRV.

These values are reported in the CCM.H-K1 CCM Key Comparison Report.

- The uncertainty of the linked Key Comparison Reference Value ($KCRV_{LINK}$) at a 95% level of confidence is calculated as in Eqn [4]:

$$U^2_{KCRV_{LINK}} = U^2_{INRiM} + U^2_{d_{CCM}} \quad (4)$$

where.

U_{INRiM}: uncertainty in INRiM's mean measurement value of this comparison.

- The deviation of UME's measurement value (d) from the associated $KCRV_{LINK}$ is calculated as in Eqn [5]:

$$d = X_{UME} - KCRV_{LINK} \quad (5)$$

where.

X_{UME}: UME's mean measurement value of this comparison.

- The uncertainty of d at a 95% level of confidence is calculated as in Eqn [6]:

$$U^2_d = U^2_{UME} + U^2_{KCRV_{LINK}} \quad (6)$$

where.

U_{UME}: expanded uncertainty in UME's mean measurement.

- The coefficient E_n is the equivalence between UME's measurements and the $KCRV_{LINK}$, and is calculated as in Eqn. (7):

$$E_n = \frac{X_{UME} - KCRV_{LINK}}{\sqrt{U^2_{UME} + U^2_{KCRV_{LINK}}}} \quad (7)$$

- The mean measurement value X_{UME} is considered equivalent to the $KCRV_{LINK}$ at 95% confidence level, if $|E_n| \leq 1$.

5.2. Measurement results not linked to the CCM.H-K1.b.c key comparison reference values (KCRVs)

The 840 HV1 comparison results cannot be linked to the CCM.H-K1 Key Comparison Reference Values due to the INRiM measurement values at the CCM KC having an E_n coefficient greater than 1. Consequently, it is not possible to have a CRV linked to the CCM KCRV for that hardness scale, and the Degree of Equivalence is calculated only between the two participants of the comparison.

- Calculation of Comparison Reference Value (CRV) for 840 HV1 Hardness:

The pilot laboratory determined the CRV for this case by calculating the weighted mean of measurements of all participants as in eqn. (8):

$$CRV = \frac{X_{INRiM}/u^2_{INRiM} + X_{UME}/u^2_{UME}}{1/u^2_{INRiM} + 1/u^2_{UME}} \quad (8)$$

- The uncertainty of the CRV is calculated by the following expression,

$$\frac{1}{u^2_{CRV}} = \frac{1}{u^2_{INRiM}} + \frac{1}{u^2_{UME}} \quad (9)$$

- Deviation of each participant from the CRV is calculated by,

$$d_{INRiM} = X_{INRiM} - CRV \quad (10)$$

$$d_{UME} = X_{UME} - CRV \quad (11)$$

- Uncertainty of the deviation at 95% level of confidence is

$$U_{d_{INRiM}} = k \times u_{d_{INRiM}} \quad (12)$$

$$U_{d_{UME}} = k \times u_{d_{UME}} \quad (13)$$

where,

$$u^2_{d_{INRiM}} = u^2_{INRiM} - u^2_{CRV} \quad (14)$$

$$u^2_{d_{UME}} = u^2_{UME} - u^2_{CRV} \quad (15)$$

and $k = 2$.

- The coefficient E_n is the equivalence between the measurements of the participating institutes and the CRV, and is calculated as follows,

$$E_{n-INRiM} = \frac{X_{INRiM} - CRV}{\sqrt{U^2_{INRiM} - U^2_{CRV}}} \quad (16)$$

$$E_{n-UME} = \frac{X_{UME} - CRV}{\sqrt{U^2_{UME} - U^2_{CRV}}} \quad (17)$$

where,

$$U_{INRiM} = k \times u_{INRiM} \quad (18)$$

$$U_{UME} = k \times u_{UME} \quad (19)$$

$$U_{CRV} = k \times u_{CRV} \quad (20)$$

and $k = 2$.

- The mean measurement value X for each participant is considered equivalent to the CRV at 95% confidence level, if the respective $|E_n| \leq 1$.

6. Comparison results

In this comparison the degree of equivalence (DoE) of UME with respect to the KCRVs of the CCM.H-K1 was calculated. Also the degree of equivalence of each participant with respect to the CRV is calculated for 840 HV1 hardness scale. These calculations comprise calculation of the

Table 3
DoE of UME wrt. the KCRV of the CCM.H-K1.b.c through INRiM in HV1 Hardness Scale.

HV1 Hardness Scale (measurement unit HV1)										
X_{UME}	U_{UME}	X_{INRiM}	U_{INRiM}	d_{CCM}	$U_{d_{CCM}}$	$KCRV_{LINK}$	$U_{KCRV_{LINK}}$	d	U_d	E_n
201.87	3.45	201.25	2.91	1.30	9.11	199.95	9.56	1.91	10.17	0.19
509.60	8.83	516.33	8.87	10.49	25.50	505.84	27.00	3.76	28.41	0.13

Table 4
DoE of INRiM and UME (wrt. the CRV) for 840 HV1 Hardness Value.

HV1 Hardness Scale (measurement unit HV1)											
X_{UME}	U_{UME}	X_{INRiM}	U_{INRiM}	CRV	U_{CRV}	d_{UME}	$U_{d_{UME}}$	E_n-UME	d_{INRiM}	$U_{d_{INRiM}}$	E_n-INRiM
848.41	18.28	833.79	15.94	840.10	12.01	8.31	13.78	0.60	-6.32	10.48	-0.60

Table 5
DoE of UME wrt. the KCRV of the CCM.H-K1.b.c through INRiM in HV30 Hardness Scale.

	HV30 Hardness Scale (measurement unit HV30)									
X_{UME}	U_{UME}	X_{INRiM}	U_{INRiM}	d_{CCM}	$U_{d_{CCM}}$	$KCRV_{LINK}$	$U_{KCRV_{LINK}}$	d	U_d	E_n
201.93	1.98	201.62	2.18	-1.32	2.45	202.94	3.28	-1.01	3.83	-0.26
506.46	6.54	507.91	5.74	-0.06	10.39	507.97	11.87	-1.51	13.55	-0.11
812.29	12.49	817.54	9.52	1.50	18.35	816.04	20.67	-3.75	24.15	-0.16

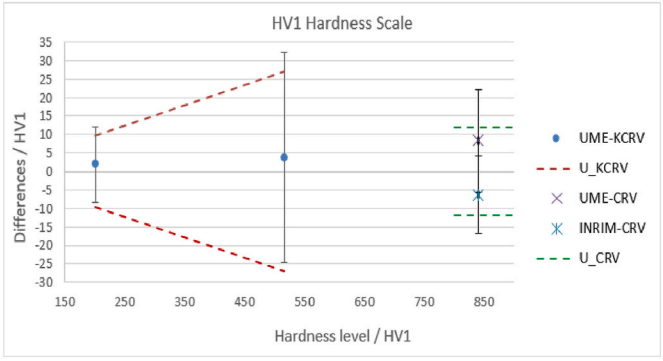

Fig. 3. Deviation of UME from the KCRV for 240 HV1 and 540 HV1, and, deviations of INRiM and UME values from the CRV for 840 HV1 with the associated expanded uncertainties.

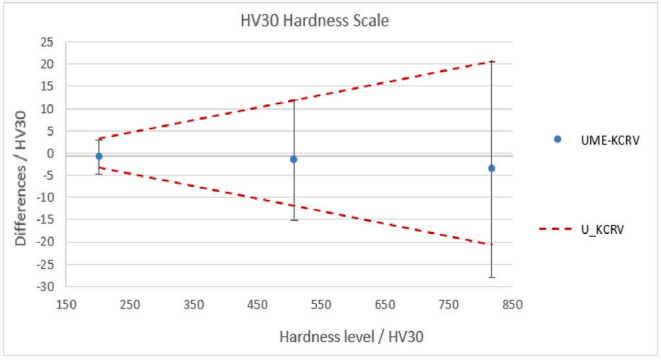

Fig. 4. Deviation of UME from the KCRV in the HV30 Hardness Scale with the associated expanded uncertainties.

deviation of each participant from the CRV, deviation of UME values from the linked KCRV and the associated uncertainties of these deviations. The E_n ratios were also calculated for each hardness scale and level. The two participants declared consistent uncertainty values and measurement results are in a significant consistency with each other and the linked KCRV. Below you can see the tabular (see Tables 3, 4 and 5) and graphical interpretation (Figs. 3 and 4) of the measurement results and calculations.

7. Summary

The EURAMET Key Comparison between INRiM and UME in the most widely used Vickers hardness scales HV1 and HV30 was completed successfully without any unexpected phenomena in any stage of it. The stability of the transfer standards during the comparison measurements was calculated and included in the measurement results.

The participating institutes declared similar uncertainty values (calculated according to [3] and [4]) and there was a significant

consistency between the measured values of the transfer standards. UME measurement results were linked to the CCM.H-K1.b.c for HV1 and HV30 scales for 240 HV, 540 HV and 840 HV values through the PL (INRiM), except for the 840 HV1.

For the measurements that could be linked to the CCM KC, the Comparison Reference Values ($KCRV_{\mathrm{LINK}}$), the UME Degrees of Equivalence (d, U_{d}) and E_{n} ratios were calculated and are shown in Tables 3 and 5 and Figs. 3 and 4 in the *Comparison Results*.

For the 840 HV1 measurements that could not be linked to the CCM KC, the CRV value was calculated using the weighted mean of the participants' measurements. The deviation values of INRiM and UME from the CRV for 840 HV1 and their uncertainties (d_{INRiM}, d_{UME}, $U_{d_{\mathrm{UME}}}$), and their E_{n} ratios were calculated and are shown in Table 4 and Fig. 3.

As a result of this comparison, DoE show a significant consistency between the UME and INRiM hardness standards, and UME with the CCM KCRV in HV1 (240 HV and 540 HV) and HV30 Vickers hardness scales and this report is supporting the present and possible new CMC submissions in the whole level of HV1 and HV30 hardness scales.

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