Result analysis of EURAMET Brinell comparison between INRiM, UME and PTB (EURAMET.M.H–S2.A.B)

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ABSTRACT

A EURAMET supplementary comparison between INRiM (National Metrology Institute of Italy), UME (National Metrology Institute of Republic of Turkey) and PTB (National Metrology Institute of Germany) had been decided to be organized in the field of Hardness Metrology to determine the consistency of the national hardness standards in these three countries realizing Brinell Hardness measurements in accordance with ISO 6506–1:2014 [1] and ISO 6506–3:2014 [2] standards. Widely used Brinell Hardness scales such as HBW 1/30 and HBW 2.5/187.5 constitute the scope of the comparison. In this paper the procedure and comparison results are explained.

1. Introduction

A supplementary comparison between INRiM (Istituto Nazionale di Ricerca Metrologica), UME (TÜBİTAK Ulusal Metroloji Enstitüsü) and PTB (Physikalisch-Technische Bundesanstalt) was carried out in the field of Hardness Metrology [1] to determine the consistency of the national hardness standards in these countries realizing Brinell Hardness measurements in accordance with the ISO 6506–1:2014 [2] and the ISO 6506–3:2014 [3] standards. The widely used Brinell Hardness scales such as HBW 1/30 and HBW 2.5/187.5 constitute the scope of the comparison which was piloted by INRiM.

The blocks to be used in the comparison were provided by UME. Each NMI realized measurement of hardness reference blocks for three hardness levels for each scale. Measurements were realized first by UME as the provider of the hardness reference blocks, then INRiM, in the same way PTB and then again by UME for checking the stability of transfer standards during the measurement period. The NMIs were requested to realize traceability of each component constituting the hardness scales on their national standards to the base SI units and constitute their uncertainty budgets before the comparison.

2. Transfer standards used

In the comparison two sets of hardness reference blocks, each set is composed of three blocks, were used for the two hardness scales of HBW 1/30 and HBW 2.5/187.5, three blocks for three hardness levels of each scale. Blocks are manufactured by YAMAMOTO SCIENTIFIC TOOL LABORATORY with a very good quality of hardness homogeneity and surface finish of test surface. Some pictures and information related to the hardness reference blocks used in the comparison are given in Fig. 1 and Tables 1 and 2.

3. Comparison procedure

Each participant first assured that the national standards used in the comparison at least were in accordance with ISO 6506–1:2014 [2] and ISO 6506–3:2014 [3] standards. Under these circumstances the components to be calibrated/verified are as follows;

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

After calibration of the components given above, the blocks were placed in the laboratory one day before the measurements for temperature equilibrium. The measurement steps made are as follows;

- Before starting the measurements, it was assured that the standardizing machines are working properly in accordance with their design parameters and relevant ISO standards requirements.
- It was assured that the anvil where the blocks are seated on and both surfaces of the reference blocks are clean.
- The ambient temperature was recorded.
- 5 hardness measurements in the marked areas on the surface of the block are made and the diameter measurement results were recorded on the data form.

4. Reference values of influence parameters

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. In this comparison the reference values were chosen according to the ISO Brinell Hardness standards specifications [2,3]. Deviations from these values were taken into the uncertainty calculations. Below, in Table 3 and in Fig. 2, the reference values and measurement cycle used in the HBW 1/30 and HBW 2.5/187.5 measurements are shown.

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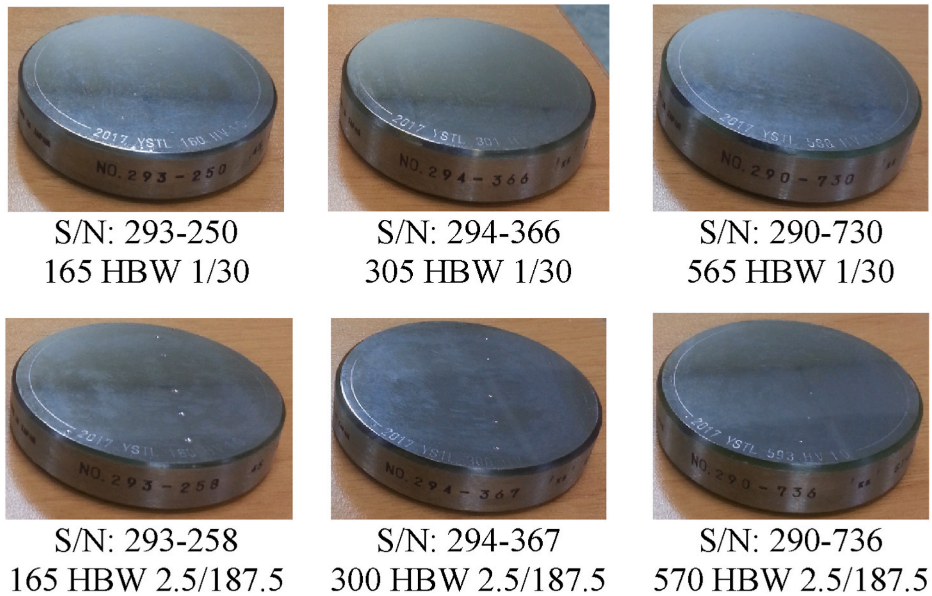

Fig. 1. Transfer standards used in the comparison.

Table 1
Hardness blocks for HBW1/30.

No	Hard. Value	Serial Number	Hardness Scale	Producer
1	165	293–250	HBW 1/30	YAMAMOTO
2	305	294–366	HBW 1/30	YAMAMOTO
3	565	290–730	HBW 1/30	YAMAMOTO

Table 2
Hardness blocks for HBW2.5/187.5

No	Hard. Value	Serial Number	Hardness Scale	Producer
1	165	293–258	HBW 2.5/187.5	YAMAMOTO
2	300	294–367	HBW 2.5/187.5	YAMAMOTO
3	570	290–736	HBW 2.5/187.5	YAMAMOTO

Table 3
Reference values for Brinell hardness Scales.

Symbol	Test parameter	Reference value	Start measurement	Stop measurement
F	Total Test force for HBW1/30	294.2 N	-	-
	Total test force for HBW2.5/187.5	1839.0 N	-	-
D	Diameter of the indenter	(1±0.003) mm (2.5±0.003) mm	-	-
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

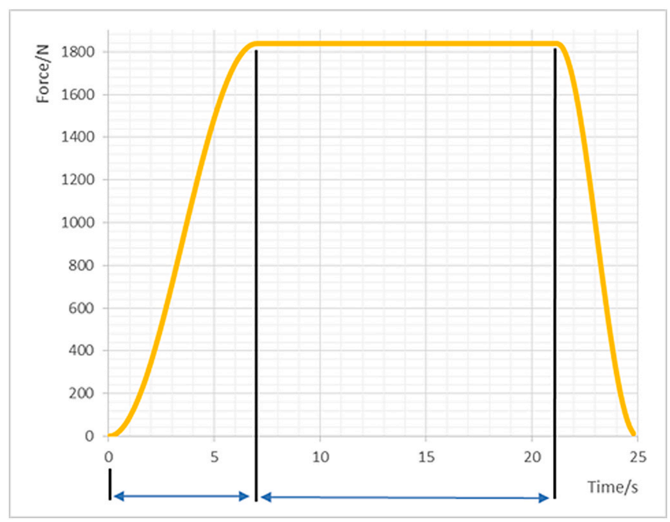

Fig. 2. Brinell Hardness Measurement Cycle (Force vs. Time).

5. Reference standards of participating institutes

- INRiM - National Metrology Institute of Italy:

A Rockwell-Brinell-Vickers hardness standard machine (INRiM-PHSM, own made and commercialized by LTF S.p.A. as GALILEO® "Primary Dead-Weight Hardness Standardazing Machines" [4]) and an indentation measurement system (own made and commercialized by LTF S.p.A. as GALILEO® GalVision [3]) are used as the national standards of Italy to provide traceability in hardness measurements in the most important and most widely used hardness scales of Rockwell, Superficial Rockwell, Brinell and Vickers hardness.

- UME - National Metrology Institute of Turkey:

A Brinell-Vickers Hardness Standard Machine (was designed by UME, produced and installed by a Turkish company İdeal Makina) and an indentation measurement system (produced by LTF S.p.A. as GALILEO® GalVision [4]) are used as the national standards of Turkey to

Table 4
DoE of INRiM, UME and PTB (wrt. The CRV) in HBW 1/30 hardness scale.

HBW 1/30 Hardness Scale							
x_{UME}	U_{UME}	x_{INRiM}	U_{INRiM}	x_{PTB}	U_{PTB}	x_{ref}	U_{ref}
166.62	1.67	165.24	1.51	172.40	1.40	168.09	4.39
301.10	3.18	300.53	1.68	309.00	2.30	303.55	5.46
571.75	6.85	574.63	6.69	595.30	4.20	580.56	14.83

HBW 1/30 Hardness Scale								
d_{UME}	U_{d_UME}	E_{n_UME}	d_{INRiM}	U_{d_INRiM}	E_{n_INRiM}	d_{PTB}	U_{d_PTB}	E_{n_PTB}
−1.47	4.69	**−0.31**	−2.85	4.64	**−0.61**	4.31	4.60	**0.94**
−2.44	6.32	**−0.39**	−3.01	5.72	**−0.53**	5.45	5.93	**0.92**
−8.81	16.34	**−0.54**	−5.93	16.27	**−0.36**	14.74	15.42	**0.96**

provide traceability in hardness measurements in the most important and most widely used hardness scales of Brinell and Vickers hardness.

- PTB - National Metrology Institute of Germany:

A Brinell-Vickers Hardness Standard Machine (VB 187,5 from the company WAZAU) and an indentation measurement system (2 dimension measuring microscope MM800 from Nikon) are used as the national standards of Germany to provide traceability in hardness measurements in the most important and most widely used hardness scales of Brinell and Vickers hardness.

All machines' specifications are all in accordance with the relevant ISO and ASTM hardness standards, particularly in regard to this comparison with the ISO 6506–1:2014 [2], the ISO 6506–3:2014 [3].

6. Institute measurement uncertainty evaluation

Before the measurements, each participant laboratory carried out the calibration of the hardness standardizing machines in order to estimate their uncertainties. The following parameters were calibrated:

- test force,
- indentation measurement system,
- diameter of the ball indenters,
- duration time of the total force,
- duration time of force application,
- reproducibility of the hardness machine.

The uncertainty budget calculations were made following the EURAMET/cg-16/v.02 [5] and the JCGM 100 [6] guidelines. Each laboratory had the responsibility for determining their own uncertainty budget and uncertainty value for each measurement.

7. Elaboration of the results

As its being a supplementary comparison, there is no possibility to assign a reference value linked to an internationally accepted reference. 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 pilot laboratory was responsible for collecting the measurement data from the participants, compiling, elaboration and preparing the reports. The measurement results were used to compute the Comparison Reference Value (CRV), degrees of equivalence (DoE) with the CRV and E_n ratios. The calculation is shown in following steps and made by making use of the following equations.

- Calculation of the Comparison Reference Value (CRV).

The pilot laboratory determined CRV by calculating the arithmetic mean of measurements of all participants (x_{ref}) by making use of the following equation,

$$x_{ref} = \frac{x_1 + x_2 + \ldots + x_n}{n} \tag{1}$$

where.

x_i: mean measured value of each participating institute, i ($i = 1, \ldots, n$), i.e, UME, INRiM, PTB and $n = 3$

- The uncertainty of the CRV (x_{ref}) was calculated by the following expression,

$$u^2(x_{ref}) = \left(\frac{STDEV(x_i)}{\sqrt{n}}\right)^2 \tag{2}$$

where.

x_i : measured value of participating institute, i ($i = 1, \ldots, n$)
$STDEV(x_i)$: standard deviation of x_i, i ($i = 1, .., n$)
$u(x_{ref})$: standard uncertainty of x_{ref}

- The deviation from the CRV is calculated by,

$$d_i = x_i - x_{ref} \tag{3}$$

- The uncertainty of this deviation at a 95% level of confidence is

Table 5
DoE of INRiM, UME and PTB (wrt. The CRV) in HBW 2.5/187.5 hardness scale.

HBW 2.5/187.5 Hardness Scale							
x_{UME}	U_{UME}	x_{INRiM}	U_{INRiM}	x_{PTB}	U_{PTB}	x_{ref}	U_{ref}
165.59	1.38	164.37	1.51	165.00	1.40	164.99	0.70
300.84	2.00	299.63	2.04	300.00	1.90	300.16	0.72
574.47	4.09	573.27	4.25	575.40	3.60	574.38	1.23

HBW 2.5/187.5 Hardness Scale								
d_{UME}	U_{d_UME}	E_{n_UME}	d_{INRiM}	U_{d_INRiM}	E_{n_INRiM}	d_{PTB}	U_{d_PTB}	E_{n_PTB}
0.60	1.55	**0.39**	−0.61	1.66	**−0.37**	0.01	1.57	**0.01**
0.68	2.13	**0.32**	−0.53	2.16	**−0.24**	−0.16	2.03	**−0.08**
0.09	4.27	**0.02**	−1.11	4.43	**−0.25**	1.02	3.81	**0.27**

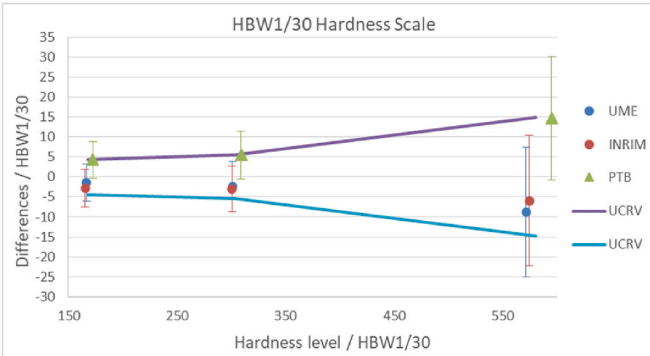

Fig. 3. Deviations of INRiM (d_{INRiM}) and UME (d_{UME}) and PTB (d_{PTB}) values from the CRV with the associated expanded uncertainty (purple and light blue lines, 95% confidence level) ($U_{d\text{-}INRiM}$ and $U_{d\text{-}UME}$ and $U_{d\text{-}PTB}$) in HBW 1/30 Hardness Scale. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

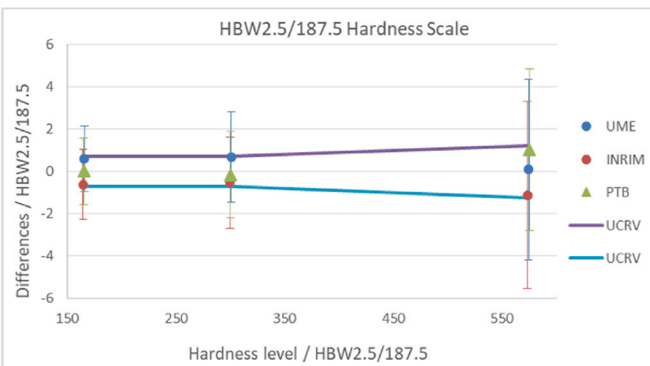

Fig. 4. Deviations of INRiM (d_{INRiM}) and UME (d_{UME}) and PTB (d_{PTB}) values from the CRV with the associated expanded uncertainty (purple and light blue lines, 95% confidence level) ($U_{d\text{-}INRiM}$ and $U_{d\text{-}UME}$ and $U_{d\text{-}PTB}$) in HBW 2.5/187.5 Hardness Scale. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

$$U(d_i) = k \cdot u(d_i) \tag{4}$$

Where $u(d_i)$ is calculated by,

$$u^2(d_i) = u^2(x_i) + u^2(x_{ref}) \tag{5}$$

and $k = 2$.

- The coefficient E_n is the equivalence between the measurements of participating institutes, is calculated as given below,

$$E_n = \frac{x_i - x_{ref}}{\sqrt{U^2(x_i) + U^2(x_{ref})}} \tag{6}$$

where,

$$U(x_i) = k \cdot u(x_i) \tag{7}$$

$$U(x_{ref}) = k \cdot u(x_{ref}) \tag{8}$$

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

x_{INRiM}	is the INRiM mean measurement value,
U_{INRiM}	is the expanded uncertainty value declared by INRiM,
x_{UME}	is the UME mean measurement value,
U_{UME}	is the expanded uncertainty value declared by UME,
x_{PTB}	is the PTB mean measurement value,
U_{PTB}	is the expanded uncertainty value declared by PTB,
x_{ref}	is the Comparison Reference Value (CRV),
U_{ref}	is the uncertainty value of CRV,
d_{INRiM}	is the deviation value of INRiM from the CRV,
U_{d_INRiM}	is the uncertainty of the deviation value of INRiM from the CRV,
E_{n_NRiM}	is the degree of equivalence of INRiM expressed in En ratio,
d_{UME}	is the deviation value of UME from the CRV,
U_{d_UME}	is the uncertainty of the deviation value of UME from the CRV,
E_{n_UME}	is the degree of equivalence of UME expressed in En ratio,
d_{PTB}	is the deviation value of PTB from the CRV,
U_{d_PTB}	is the uncertainty of the deviation value of PTB from the CRV,
E_{n_PTB}	is the degree of equivalence of PTB expressed in En ratio.

8. Comparison results

In this comparison the degree of equivalence of each participant with respect to the CRV was calculated. This calculation comprises calculation the deviation of each participant from the CRV and the associated uncertainty of this deviation. Also the E_n ratios were also calculated for each hardness scale and level. The three participants declare consistent uncertainty values and measurements results are in a significant consistency with each other. In Tables 4 and 5 and in Figs. 3 and 4, tabular and graphical interpretation of the measurement results and calculations are shown.

In regard to the calculations shown in Tables 4 and 5, the graphical representations are shown in Figs. 3 and 4.

9. Summary

At the end of the supplementary comparison between INRiM, UME and PTB in the most widely used Brinell hardness scales HBW 1/30 and HBW 2.5/187.5 it was completed without any unexpected phenomena in any stage of it. The participating institutes declared similar uncertainty values and there was a significant consistency between the measured values of the transfer standards. The comparison reference values (CRVs), the deviation value of INRiM, UME and PTB from the CRV and their uncertainties (d_{INRiM}, d_{UME} d_{PTB}, $U_{d_{INRiM}}$, $U_{d_{UME}}$, $U_{d_{PTB}}$), and their E_n ratios were calculated and shown in Tables 4 and 5 and Figs. 3 and 4 in the *Comparison Results*. The degrees of equivalence show a significant consistency between INRiM, UME and PTB hardness standards in HBW 1/30 and HBW 2.5/187.5 Brinell hardness scales and this comparison is supporting the present and possible new CMC submissions.

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