EURAMET Supplementary Comparison between INRiM, UME and PTB in Brinell Hardness Scales (HBW1/30 – HBW2.5/187.5)

Final Report

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Abstract

This report describes a EURAMET supplementary comparison in Brinell hardness scales of three national metrology institutes of Italy, Turkey and Germany; INRiM, UME and PTB, respectively. The pilot laboratory is INRiM in the comparison where two sets of hardness reference blocks for the Brinell Hardness scales of HBW1/30 and HBW2.5/187.5 were used. Each set of blocks consists of three hardness levels used for each of the two scales. The comparison was realized as planned in the Technical Protocol with some delay. The measurement results and uncertainty assessments declared by INRiM, UME and PTB are in consistency with each other.

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 to determine the consistency of the national hardness standards in these countries realizing Brinell Hardness measurements in accordance with the ISO 6506-1:2014 [1] and the ISO 6506-3:2014 [2] standards. The widely used Brinell Hardness scales such as HBW1/30 and HBW2.5/187.5 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 each of the two scales on the same transfer standards. Measurements were carried out first by UME as the provider of the blocks, then by the pilot laboratory INRiM, then by PTB and again by UME for checking the stability of the transfer standards during the measurement period.

The NMIs were requested to realize the 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 measurements.

2. Participating Institutes

Organizer	:	The European Association of National Metrology Institutes, **EURAMET**
Pilot Lab.	:	**Alessandro GERMAK** **INRiM** – Istituto Nazionale di Ricerca Metrologica Strada delle Cacce, 91 10135 Torino, **ITALY** Tel: +39 011 3919 924 a.germak@inrim.it
Participating Lab.	:	**Cihan KUZU** **UME** – TÜBİTAK Ulusal Metroloji Enstitüsü TÜBİTAK Gebze Yerleşkesi

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Participating Lab.	**:**	**Febo MENELAO** **PTB** – Physikalisch-Technische Bundesanstalt Bundesallee 100 38116 Braunschweig, **GERMANY** Tel: +49 531 592-5145 febo.menelao@ptb.de

3. 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. The 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 [1], the ISO 6506-3:2014 [2]. The specifications of the machines related to Brinell hardness are as mentioned below.

Scales realized	:	Brinell scales constituted with the force values of 5 kgf, 6.25 kgf, 10 kgf, 15.625 kgf, 30 kgf, 31.25 kgf, 62.5 kgf, 187.5 kgf and relevant Tungsten Carbide ball indenters.
Indenters	:	1 mm, 2.5 mm and 5 mm Tungsten Carbide ball indenters
Force application	:	Dead weight force application system
Measurement cycle	:	Laser interferometer optic system and force measurement sensor
Indentation Measurement Sys.	:	An optical microscope with a special software prepared for indentation measurements for Brinell and Vickers
Operation	:	PC Controlled, special software, automatic testing cycle and hardness indentation realization and measurement.
Calibration procedure	:	Machines are able to carry out hardness reference block calibration according to ISO 6506-1 [1], ISO 6506-3 [2] and ASTM E-10 [3] Standards for Brinell scales.

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 provide traceability in hardness measurements in the most important and most widely used hardness scales of Brinell and Vickers hardness. The 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 [1], the ISO 6506-3:2014 [2]. The specifications of the machine related to Brinell hardness are as mentioned below.

Scales realized	:	Brinell scales constituted with the force values of 5 kgf, 6.25 kgf, 10 kgf, 15.625 kgf, 30 kgf, 31.25 kgf, 62.5 kgf, 187.5 kgf, 250 kgf and relevant Tungsten Carbide ball ind.
Indenters	:	1 mm, 2.5 mm and 5 mm Tungsten Carbide ball indenters
Force application	:	Dead weight force application system
Measurement cycle	:	Force measurement sensor
Indentation Measurement Sys.	:	An optical microscope with a special software prepared for indentation measurements for Brinell and Vickers
Operation	:	PC Controlled, special software, automatic testing cycle and hardness indentation realization and measurement.
Calibration procedure	:	Machines are able to carry out hardness reference block calibration in accordance with the ISO 6506-1 [1], the ISO 6506-3 [2] and the ASTM E-10 [3] Standards for Brinell scales.

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. The 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 [1], the ISO 6506-3:2014 [2]. The specifications of the machines related to Brinell hardness are as mentioned below.

Scales realized	:	Brinell scales constituted with the force values of 15.625 kgf, 25 kgf, 30 kgf, 31.25 kgf, 62.5 kgf, 125 kgf, 187.5 kgf and relevant Tungsten Carbide ball indenters.
Indenters	:	1 mm, 2.5 mm and 5 mm Tungsten Carbide ball indenters
Force application	:	Dead weight force application system
Measurement cycle	:	Hydraulic movment system with elecronic detection of the phases of the system
Indentation Measurement Sys.	:	An optical microscope with a length measuring software prepared for indentation measurements for Brinell and Vickers
Operation	:	Electronic controlled, automatic testing cycle and hardness indentation realization
Calibration procedure	:	Machines are able to carry out hardness reference block calibration according to ISO 6506-1 [1], ISO 6506-3 [2] and ASTM E-10 [3] Standards for Brinell scales.

4. Transfer Standards Used in the Comparison

In the comparison two sets of hardness reference blocks were used, one set for each hardness scale of HBW1/30 and HBW2.5/187.5, separately. Three blocks for each scale, a total of 6 hardness reference blocks were used. Information related to the transfer standards used in the comparison is given in Tables 1 and 2.

Table 1. Hardness blocks for HBW1/30 Scale.

No	Hardness Value	Serial Number	Producer
1	165	293-250	YAMAMOTO
2	305	294-366	YAMAMOTO
3	565	290-730	YAMAMOTO

Table 2. Hardness blocks for HBW2.5/187.5 Scale.

No	Hardness Value	Serial Number	Producer
1	165	293-258	YAMAMOTO
2	300	294-367	YAMAMOTO
3	570	290-736	YAMAMOTO

The first measurements were carried out by UME as the provider of the blocks, then the transfer standards were sent to the pilot laboratory INRiM to perform their measurements, and then in the same way to PTB, furtherly, the hardness blocks were

sent back to UME. Then UME performed its second measurements for checking the stability of the transfer standards. Pictures of the transfer standards used in the comparison are shown in Figures 1 and 2.

| S/N: 293-250, 165 HBW1/30 | S/N: 294-366, 305 HBW1/30 | S/N: 290-730, 565 HBW1/30 |

Figure 1. Transfer Standards for HBW1/30 Scale.

| S/N: 293-258, 165 HBW2.5/187.5 | S/N: 294-367, 300 HBW2.5/187.5 | S/N: 290-736, 570 HBW2.5/187.5 |

Figure 2. Transfer Standards for HBW2.5/187.5 Scale.

5. Procedure

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

- Force
- Measurement cycle (in accordance with the ISO 6506-1:2014 [1] and the ISO 6506-3:2014 [2] standards).
- Indenter (in accordance with the ISO 6506-3:2014 [2] standard).
- Indentation measurement system (in accordance with the ISO 6506-3:2014 [2] standard).

After 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 are as follows;

- Before starting the measurements, it was assured that the standardizing machines were working properly in accordance with their design parameters and relevant ISO Hardness Standards requirements.
- Assured that the anvil where the blocks are seated on and both surfaces of the reference blocks were clean.
- Assured that 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 and recorded on the data form.
- The ambient temperature was recorded.

6. 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 [1] and [2]. Deviations from these values were taken into the uncertainty calculations. Table 3 gives the reference values of the influence parameters and Figure 3 illustrates the measurement cycle used in the HBW1/30 and HBW2.5/187.5 measurements.

Table 3. Reference Values for Brinell Hardness Scales HBW1/30 and HBW2.5/187.5.

| 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 (see Figure 3)	(7±1) s	~1% F	~99% F
T_d	Duration of the total force (see Figure 3)	(14±1) s	~99% F	~99% F
T	Temperature of test	23°C	Beginning of the test	End of the test
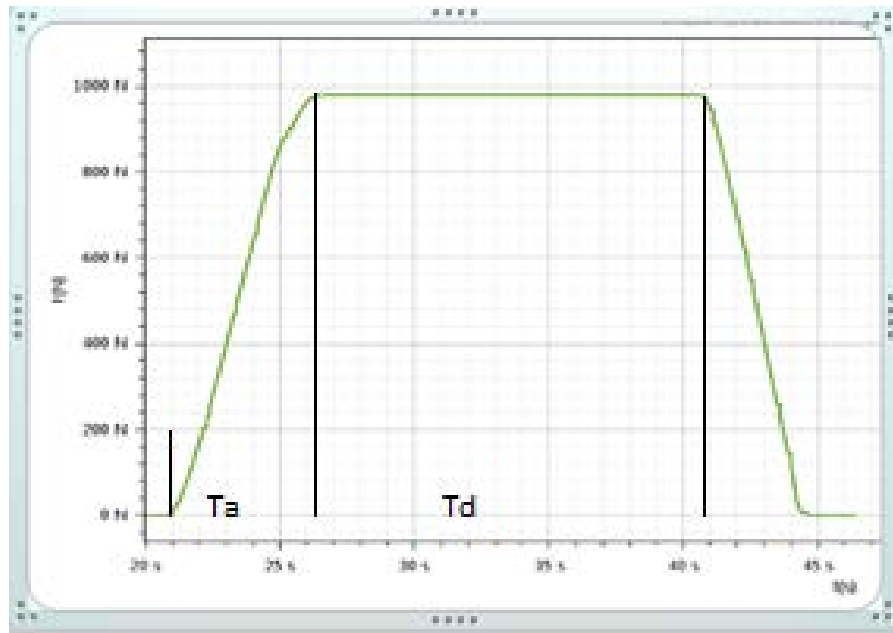
Figure 3. Identification of the Brinell Hardness Measurement Cycle.

7. 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.

8. Time Table of the Measurements

The measurements were made first by UME as the provider of the reference hardness blocks, then by the pilot laboratory INRiM and finally by UME again for checking the stability of the transfer standards. UME's mean value of the first and second set of measurements is used to compute the degree of equivalence with the Comparison Reference Value (CRV) and E_n ratio. The measurement schedule is given in Table 4.

Table 4. Schedule of the measurements.

Institute/Country	Lab	Date of measurements
UME, Turkey	Participant	September, 2019
INRiM, Italy	Pilot	October, 2019
PTB, Germany	Participant	November, 2019
UME, Turkey	Participant	December, 2019

9. Transportation

The travelling standards (hardness reference blocks) were transported in a wooden box protective case which was prepared by UME. When the blocks were sent/transported the receiving institute checked them and notified the sending laboratory by email. Before packing the blocks for transportation to the next participant (after measurements are finished) they were cleaned and wrapped in their anti-rusting paper, put in their original case and placed in the transportation box in order to prevent any damage during transportation.

10. Data Compilation

Each laboratory entered 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 and preparing the reports.
The results will be used to support the CMCs of the participating laboratories declared in Appendix C of the MRA.

11. Data Elaboration

The measurement results are used to compute the degree of equivalence with Comparison Reference Value (CRV) and E_n ratio. The calculation is shown in following steps and made by making use of the following equations.

- Calculation of 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,

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

where,

x_i : mean measured value of each participating institute, i ($i = 1, ..., n$), 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,

$STDEV(x_i)$: 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

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

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 x_i is equivalent with x_{ref} (CRV) at 95% confidence level, if $|E_n| \leq 1$,

    where,

    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 E_n 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 E_n 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 E_n ratio.

12. Stability of the Transfer Standards

The stability of hardness reference blocks during the comparison measurements was calculated as the difference between the first and second measurements made by UME as the blocks provider. Except for the 570 HBW1/30 where the difference could be considered as significant (no specific reasons have been founded), for all other measurements there was no significant deviation as seen in Tables 5 and 6. The deviation was taken into account in the measurement results by calculating its contribution to the uncertainty of the mean value accepting it as a rectangular distribution ($\frac{deviation}{2\sqrt{3}}$).

Table 5. Stability of the Transfer Standards in HBW1/30 Scale.

Scale	Block S. N.	Measurement Values		Non Uniformity / %		Deviation/ HBW1/30	Mean Value	Uncert./ HBW1/30
		1st Meas.	2nd Meas.	1st Meas.	2nd Meas.			
HBW1/30	293-250	166.66	166.58	0.65	0.96	-0.08	166.62	1.67
	294-366	301.36	300.85	0.32	0.30	-0.51	301.10	3.18
	290-730	570.49	573.00	0.15	0.17	2.51	571.75	6.85

Table 6. Stability of the Transfer Standards in HBW2.5/187.5 Scale.

Scale	Block S. N.	Measurement Values		Non Uniformity / d%		Deviation/ HBW2.5/ 187.5	Mean Value	Uncert./ HBW2.5/ 187.5
		1st Meas.	2nd Meas.	1st Meas.	2nd Meas.			
HBW 2.5/187.5	293-258	165.56	165.63	0.65	0.41	0.07	165.59	1.38
	294-367	301.09	300.60	0.27	0.05	-0.49	300.84	2.00
	290-736	574.51	574.42	0.35	0.09	-0.10	574.47	4.09

13. Comparison Results

The EURAMET Supplementary Comparison between INRiM, UME and PTB in the widely used Brinell hardness scales HBW1/30 and HBW2.5/187.5 was completed 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 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 (X_{ref}) and their uncertainties; d_{INRiM}, d_{UME}, d_{PTB}, U_{d_INRiM}, U_{d_UME}, U_{d_PTB}, and E_n ratios were calculated and shown in Tables 7 and 8 and Figures 4 and 5 in the Annex. The degrees of equivalence show a significant consistency between INRiM, UME and PTB hardness standards in HBW1/30 and HBW2.5/187.5 Brinell hardness scales and this report is supporting the present and possible new CMC submissions.

14. References

[1] EN ISO, 6506-1:2014. Metallic Materials – Brinell Hardness Test – Part1: Test Method.

[2] EN ISO, 6506-3:2014. Metallic Materials – Brinell Hardness Test – Part3: Calibration of Reference Blocks.

[3] ASTM E-10:2018. Standard Test Method for Brinell Hardness of Metallic Materials

[4] http://www.ltf.it/en/prodotti.php?b=3&c=636&p=3361

[5] EURAMET/cg-16/v.02, Guidelines on the Estimation of Uncertainty in Hardness Measurements, 2011.

[6] JCGM 100: 2008, Evaluation of measurement data - Guide to the expression of uncertainty in measurement.

Annex: Measurement Data

Table 7. Degree of Equivalence of INRiM, UME and PTB (wrt. the CRV) in HBW1/30 Hardness Scale.
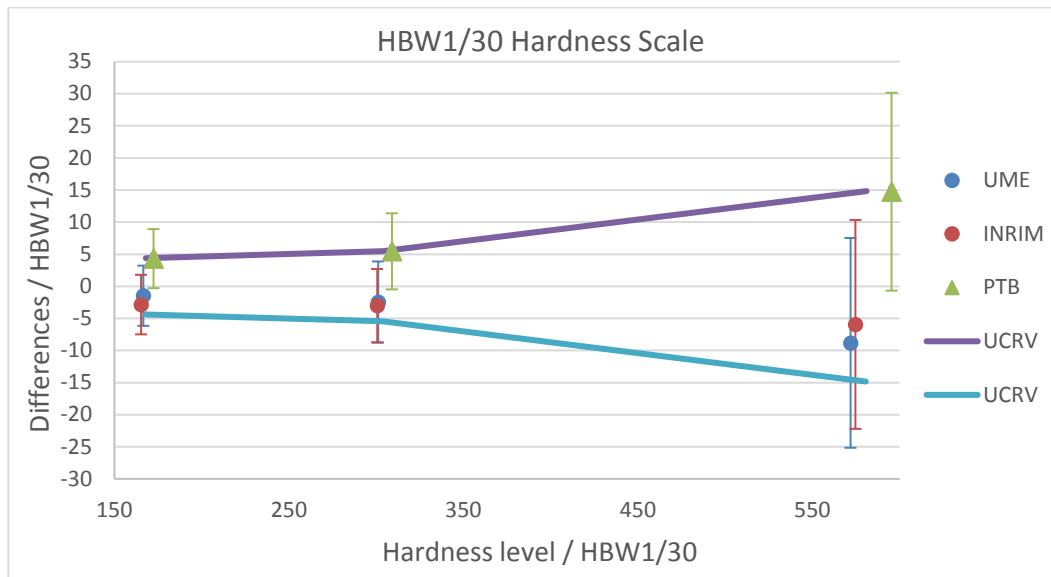
HBW1/30 Hardness Scale							
x_{UME}	U_{UME}	x_{INRiM}	U_{INRiM}	x_{PTB}	U_{PTB}	x_{ref}	U_{ref}
166.62	1.67	165.24	1.51	172.40	1.40	168.09	4.39
301.10	3.18	300.53	1.68	309.00	2.30	303.55	5.46
571.75	6.85	574.63	6.69	595.30	4.20	580.56	14.83

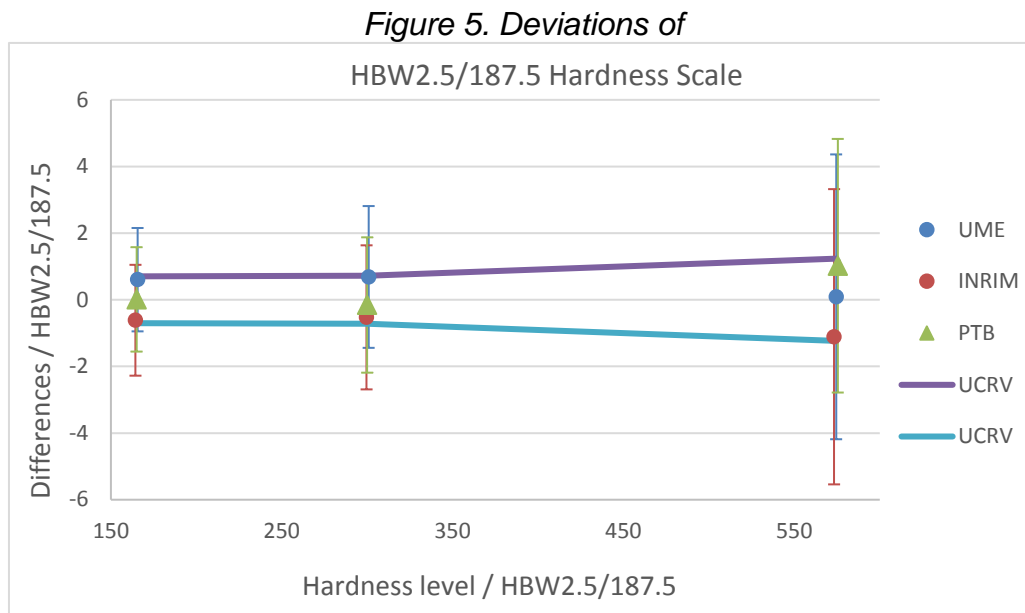| HBW1/30 Hardness Scale								
d_{UME}	U_{d_UME}	E_{n_UME}	d_{INRiM}	U_{d_INRiM}	E_{n_INRiM}	d_{PTB}	U_{d_PTB}	E_{n_PTB}
-1.47	4.69	-0.31	-2.85	4.64	-0.61	4.31	4.60	0.94
-2.44	6.32	-0.39	-3.01	5.72	-0.53	5.45	5.93	0.92
-8.81	16.34	-0.54	-5.93	16.27	-0.36	14.74	15.42	0.96

Table 8. Degree of Equivalence of INRiM, UME and PTB (wrt. the CRV) in HBW2.5/187.5 Hardness Scale.

HBW2.5/187.5 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

HBW2.5/187.5 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

Figure 4. Deviations of INRiM (d_{INRiM}) and UME (d_{UME}) and PTB (d_{PTB}) values from the CRV with the associated expanded uncertainty (95% confidence level) ($U_{d-INRiM}$ and U_{d-UME} and U_{d-PTB}) in HBW1/30 Hardness Scale.

Figure 5. Deviations of

INRiM (d_{INRiM}) and UME (d_{UME}) and PTB (d_{PTB}) values from the CRV with the associated expanded uncertainty (95% confidence level) ($U_{d-INRiM}$ and U_{d-UME} and U_{d-PTB}) in HBW2.5/187.5 Hardness Scale.