UDC 006.91:621.753.38:531

V.V. Skliarov

National Scientific Centre "Institute of Metrology", Kharkiv, Ukraine

REGIONAL METROLOGICAL ORGANIZATION KEY COMPARISON IN THE FIELD OF ROCKWELL HARDNESS SCALES (PROGRESS AND PERSPECTIVES)

In light of the new Rockwell C hardness scale definition adopted by National Metrology Institutes (NMIs) [1], a comparison of the Hardness Rockwell and Superficial- Rockwell scales realized by National primary hardness Rockwell machines is necessary to verify the implementation of the new definition. A Technical Protocol of a Regional Metrological organization Key Comparison (RMO KC) was presented and approved at the t TC 1.6 meeting in September 2014 in Braunschweig, Germany. A Regional Key Comparison (RKC) has been chosen and the technical protocol has been prepared following the rules given by the document "Measurement comparisons in the CIPM MRA" [2].

Keywords: *key comparison, hardness, Rockwell scales, National primary standards, reference blocks, measurement area, budget of uncertainty, uncertainty evaluation.*

Introduction

The aim of the regional key comparison is to extend the metrological equivalence over the measurement standards of national metrology institutes, which did not take part in CIPM KC [3].

The degree of equivalence of measurement standards of the NMIs participating in key comparisons of a Regional Metrology Organization (RMO), is determined in accordance with section T4 of the Technical Supplement to the Mutual Recognition Arrangement (MRA) with respect to a reference value of CIPM KCRV, using the results of measurements obtained at the NMIs that participated in both comparisons (linking NMIs).

The procedures used for RMO KC data evaluation is intended to provide linking to CIPM KC data with low uncertainty and they should correspond to those used for CIPM KC data evaluation.

During the 17th meeting of the Technical committee (TC 1.6) in September 2012 in Azerbaijan it was agreed to carry out the RMO KC of the Rockwell scales in which the hardness laboratories of National Metrology Institutes should participate. In the following meeting of the TC 1.6 in October, 2013 in Ukraine, the details of the Regional Key Comparison (RKC) organization was discussed.

The National Scientific Centre "Institute of Metrology" (NSC "IM") - pilot laboratory decided to purchase the test blocks required for the comparison from a single source to eliminate any question of a block bias due to using blocks from different manufacturers. The RMO KC requires each NMI to test 5 (five) hardness levels using their own indenter. The proposed 5 (five) hardness levels are 80-86 HRA, 80-100 HRBW, 20-30 HRC, 40-50 HRC and 60-70 HRC. It was determined that one set of blocks will be required for the Regional Key Comparison.

1. Program of measurements

For the RMO KC, there will be 1 set of hardness reference blocks of the Rockwell scale, consisting of 5 (five) blocks with the hardness levels 80-86 HRA, 80-100 HRBW, 20-30 HRC, 40-50 HRC, 60-70 HRC (Fig. 1). Hardness blocks are manufactured by "Centre "MET" Ltd (Russia) and have a length of 60 mm, width of 40 mm and a thickness of 6 mm. The upper surface of the block, which is the measurement surface, is finished. The measurement area is defined to be within an engraved grid. A grid (7 mm × 7 mm) is engraved on the block surface in order to define the coordinates of the test locations. The sizes of the test area and grid allow 35 possible test locations. (Fig. 2).

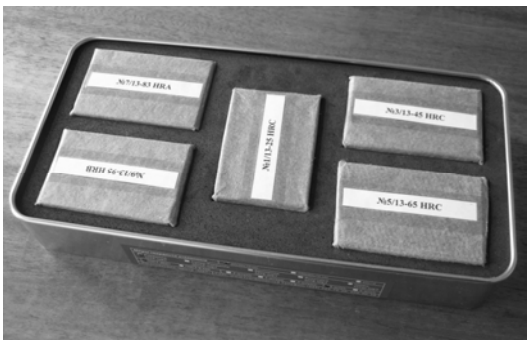

Fig. 1. Rockwell's set of hardness reference blocks

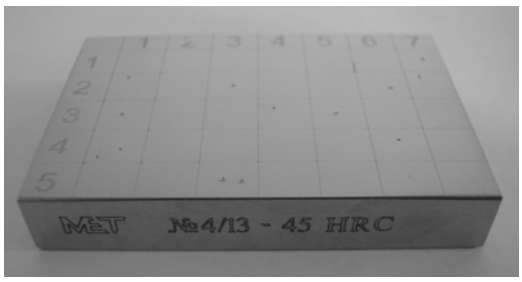

Fig. 2. Layout of the grid on the measurement surface of the hardness reference blocks with the logo

The pilot laboratory are responsible for purchasing the blocks for their regional comparison, while each participating institute bears the costs for transport, customs and related administrative fees. The pilot laboratory is to make measurements at the beginning and at the end of the RKC in order to evaluate the stability of the hardness reference blocks used in the RKC.

2. Procedure

The measurements shall be carried out following the Rockwell scale definition developed for adoption by National Metrology Institutes (NMIs). Any deviations from the procedure must be reported. Before conducting the measurements, each participant shall carry out the calibration of the primary hardness machine. Results of the calibration are:

- measurement results and uncertainties of the test forces (F_0, u_{F0} and F, u_F),
- uncertainty of the length measuring system u_l,
- measurement results and uncertainties of the indenter geometry (plane angle α_m, $u_{\alpha m}$, spherical tip radius R_a, u_{Ra}),
- measurement results and uncertainties of the indenter geometry (spherical tip diameter R_β, $u_{R\beta}$),
- measurement result and uncertainty of the final indentation speed of additional load application (V_{fis}, u_{Vfis}),
- measurement results and uncertainties of the total time of preliminary test force (T_p, u_{Tp}),
- application time of preliminary test force (T_a, u_{Ta}),
- duration time of preliminary test force (T_{pm}, u_{Tpm}),
- duration of the total force (T_{df}, u_{Tdf}),
- final reading time (T_{rf}, u_{Trf}),
- measurement result and uncertainty of the temperature of test (T, u_T).

Each participant shall make 5 (five) measurement indentations on each hardness reference block. The 5 (five) indentations will be used to evaluate the stochastic deviations occurring during the measurements, including the evaluation of the inhomogeinity of the hardness distribution across the test surface of the hardness reference blocks. The example of the test locations on the blocks will be provided to participants as grid coordinates (Fig. 3).

	1	2	3	4	5	6	7
1	**U**	**B**	**G**	**B**	**G**	**U**	**C**
2	**C**	**U**	**K**	**C**	**K**	**B**	**G**
3	**B**	**G**	**C**	**U**	**B**	**C**	**K**
4	**U**	**K**	**B**	**G**	**K**	**U**	**G**
5	**G**	**C**	**K**	**U**	**B**	**C**	**K**

Fig. 3. Test locations on the reference block

For each participant lab was determined symbol (see Table 1). It will be used by the participants to find the location of the indentations on the hardness block surface (1-5), as indicated in Fig. 3. As additional locations (6 and 7), for test measurement or in case a measurement error occurs and the measurement must be repeated, the locations indicated as "repeat measurements" can be used.

Each indentation shall be made at the center of the open square within the engraved grid lines. An indentation must not contact an engraved line. If a measurement error occurs, participant register the error in the data report in the field for measurement errors (designation "error") and repeat the measurement at one of the unused locations designated for repeat measurements.

After completing all measurements, participant shall clean the blocks with alcohol, then wrap the blocks in anti-corrosion paper and put them in a plastic bag. The blocks are to be securely packed in the original shipping container and shipped to the next participant. When the blocks are shipped, the sending institute must again notify the pilot laboratory by fax or email.

Due to the number of fields on the hardness block surface (35) and the number of indentations to be carried out by each laboratory (5 plus two), the maximum number of participants for each regional comparison is 5 (35/5=5+2 for possible errors or test indentation). The Pilot laboratory must perform the measurements twice. After RSE "KazInMetr", re-indentation for the pilot laboratory will be performed in the free zones.

3. Time table of the measurements

Table 1

Time table of the measurements

The symbol of the cell	Institute/Country
U	NSC "IM", **U**kraine
G	PTB, **G**ermany
C	CMI, **C**zech Republic
B	BelGIM, **B**elarus
K	RSE "KazInMetr", **K**azakhstan
U	NSC "IM", **U**kraine

4. Uncertainty evaluation

"A result from a participant is not considered complete without an associated uncertainty, and is not included in the draft report unless it is accompanied by an uncertainty supported by a complete uncertainty budget. Uncertainties are drawn up following the guidance given in the technical protocol" [2].

The uncertainty in measurement should be estimate according to the GUM [3, 5]. At least the following influence quantities on the uncertainty should be considered:

- test forces;
- depth measuring system;

- cone angle of the indenter;
- spherical tip of the indenter;
- final indentation speed of additional force application;
- total time of preliminary test force;
- duration of the total force;
- final reading time;

reproducibility of the primary hardness machine.

The draft of EURAMET/cg-16/v.01 (previous EA-10/16) [5, 6] may be used. For the uncertainty budget calculation and for the sensitivity coefficients an example is given in Table 2. Each laboratory has the responsibility for determining their own uncertainty budget, including the determination of the sensitivity coefficients.

5. Data compilation

Pilot Laboratory is responsible for collecting and compiling the measurement data from participants.

The first Draft (Draft A), should be prepared as soon as all the results have been received from the participants. It includes the results transmitted by the participants, identified by name. It is confidential to the participants.

After comments, remarks and discussion, the second Draft (Draft B), should be subsequently prepared for the pilot laboratory and will include the Appendix containing proposals for a reference value and degrees of equivalence.

According to the [7] we have two procedures of data evaluation designated by "C" and "D" which conventionally correspond to two types of comparison and ways for RMO KC data transformation. The "C" procedure can be applied to those comparisons that require determination of a physical quantity value and assignment of this value to the material measure.

In the appropriate CIPM and RMO key comparisons, the material measures with close but, nevertheless, different values of a physical quantity can be used as travelling standards. The "C" procedure requires application of an additive correction for RMO KC data. It is assumed that measurement uncertainties associated with the results of linking NMIs, obtained in the CIPM KC and RMO KC, remain the same. It is our case. It is important to take into account that in comparisons take part institutions with CMC line in BIPM. This must be taken into account when calculating the additive correction (correction estimate in case of a two of linking NMIs – PTB and RSE "KazInMetr").

The Draft B should be presented and discussed at the next meeting TC 1.6 for agreement. After agreement Draft B should be presented to CCM-WGH. If it is approved by the CCM-WGH, the Final Report will be prepared and sent to the CCM.

Details of the procedure on the preparation of the RKC reports are given in [2, 6]. Please note the following point:

"If, on examination of the complete set of results, the pilot institute finds results that appear to be anomalous, the corresponding institutes are invited to check their results for numerical errors but without being informed as to the magnitude or sign of the apparent anomaly. If no numerical error is found, the result stands, and the complete set is sent to all participants".

6. Perspectives

In carrying out comparisons on scales Superficial-Rockwell use measures with a large number of zones for indentation. This will allow a greater number of participating NMI. Subject to availability on measures to give the status of comparisons «on-going». At presence of a second set of measures of hardness, with the participants agree on the use of the general indenter during comparisons Superficial-Rockwell.

Table 2

Example of evaluation of the uncertainty based
on the state of the art of primary hardness standard machines for the 80-86 HRA hardness level

Parameter/ Unit	Difference from the nominal value, Δxi	Standard uncertainty, $u(x_i) = \Delta x_i/\sqrt{3}$	Sensitivity coefficient $c_i = \Delta H_i/\Delta x_i$	Variation in hardness, HRA, $\Delta H_i = \Delta x_i \times c_i$	Contribution to the uncertainty, $u_{i2}(H)$,HRA_2
F_0, N	0,2	0,115	0,056	0,0112	0,006
F, N	1,5	0,866	-0,041	-0,0616	-0,036
α_m, °	0,1	0,058	0,15	0,015	0,009
R_a, mm	1,0	0,577	0,15	0,15	0,087
l, (μm)	0,1	0,058	0,5	0,05	0,029
V_{fis}, (μm/s)	0,7	0,404	-0,0064	-0,0448	-0,026
T_p, s	0,2	0,115	-0,01	-0,002	-0,001
T_{df}, s	0,2	0,115	-0,01	-0,002	-0,001
d, μm	0,3	0,173	0,5	0,15	0,087
Total				0,266	0,154
Standard uncertainty u_m, HRA					0,134
Degrees of freedom (Welch-Satterthwaite formula)					64
Coverage factor **k** for confidence level p = 95%, (t-Student)					2
Expanded uncertainty $U = k \cdot u_m$, HRA					0,268

Conclusion

During international comparisons, the calculated values of the expanded uncertainty are compared with the values of the expanded uncertainty of the participating countries of international comparisons. For each sub band, the hardness are plotted showing the measured each participant comparisons hardness values with expanded uncertainty.

As an example, in Fig. 4 shown a graphical representation of the measurement result for the sub-band 60-70 HRC (66 HRC) for a 5 NMI - comparisons of participants. According to the results of the graphical representation of the results it is concluded confirmation or non-confirmation of the declared values of uncertainty for each participant for each of the sub hardness.

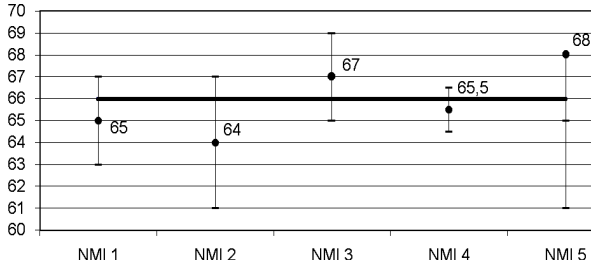

Fig. 4. The results of international comparisons of the reference point 66 HRC (an example)

The positive results of international comparisons allow participating countries confirm the implementation of the "Agreement on mutual recognition of National measurement standards and calibration and measurement certificates issued by the National metrology institutes» and get the right to submit the appropriate line CMC (calibration and measurement capabilities) for calibration and measurement capabilities of the country on the website JCRB (Joint Committee of the Regional Metrology Organizations and the BIPM) - Joint Committee of the International Bureau of Weights and Measures (BIPM) and the RMO.

References list

1. HRC Definition [Электронный ресурс]. – Режим доступа: http://www.bipm.org/wg/CCM/CCM-WGH/Allowed/International_definitions/HRC_definition.pdf.

2. Measurement comparisons in the CIPM MRA. [Электронный ресурс]. – Режим доступа: http://www.bipm.org/utils/common/CIPM_MRA/CIPM_MRA-D-05.pdf.

3. Bureau International des Poids et Mesures (BIPM) 1999 Mutual recognition of national standards and of calibration and measurement certificates issued by national metrology institutes BIPM Publication (Sevres:BIPM).

4. JCGM 100:2008 (BIPM, IEC, IFCC, ILAC, ISO, IUPAC, IUPAP and OIML), GUM 1995 with minor corrections, Evaluation of measurement data — Guide to the expression of uncertainty in measurement. [Электронный ресурс]. – Режим доступа: http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf/

5. EURAMET/cg-16/v.01 (Previously EA-10/16), "Guidelines on the Estimation of Uncertainty in Hardness Measurements", July 2007. [Электронный ресурс]. – Режим доступа: http://www.euramet.org/index.php?eID=tx_nawsecuredl&u=0&file=fileadmin/docs/Publications/calguides/EURAMET-cg-16.01_Hardness.pdf&t=1221740315 &hash=d5e4723b7d3540ec2110dfe6376ffef0.

6. Brice L. Uncertainty in hardness measurement, NPL Report CMAM 87, April 2003 [Электронный ресурс] / L. Brice, F. Davis, A. Crawshaw. – Режим доступа: (http://www.npl.co.uk/engineering-measurements/mass-force-pressure/hardness/rockwell-hardness-sensitivity-coefficients).

7. COOMET R/GM/14:2006 Guidelines for data evaluation of COOMET key comparison [Электронный ресурс]. – Режим доступа: http://www.coomet.org/EN/doc/r14_2006.pdf.

Поступила в редколлегию 16.12.2014

Рецензент: д-р техн. наук, проф. Ю.П. Мачехин, Харьковский национальный университет радиоэлектроники, Харьков.

КЛЮЧОВІ ЗВІРЕННЯ РЕГІОНАЛЬНИХ МЕТРОЛОГІЧНИХ ОРГАНІЗАЦІЙ (ХІД ВИКОНАННЯ ТА ПЕРСПЕКТИВИ)

В.В. Скляров

У статті відображено алгоритм виконання робіт з міжнародних ключових звірень регіональних метрологічних організацій КООМЕТ. Описано використання мір твердості при проведенні вимірювань за участю 5 метрологічних інститутів. Розглянуто питання складання бюджету невизначеності з використанням коефіцієнтів чутливості. Запропоновано, в якості перспективних робіт, проведення звірень за шкалами Супер-Роквелла. Надання статусу звірень on-going, у випадку наявності вільних місць для індентування, після основного кола звірень, на мірах твердості.

***Ключові слова**: ключові звірення, шкалі Роквелла, Державний первинний еталон, міри твердості, область вимірювань, бюджет невизначеності, оцінка невизначеності.*

КЛЮЧЕВЫЕ СЛИЧЕНИЯ РЕГИОНАЛЬНЫХ МЕТРОЛОГИЧЕСКИХ ОРГАНИЗАЦИЙ (ХОД ВЫПОЛНЕНИЯ И ПЕРСПЕКТИВЫ)

В.В. Скляров

В статье представлен алгоритм выполнения работ при проведении международных ключевых сличений региональных метрологических организаций КООМЕТ. Описано использование меры твердости при проведении измерений с участием 5 метрологических институтов. Рассмотрен вопрос составления бюджета неопределенности с использованием коэффициентов чувствительности. Предложено, в качестве перспективных работ, проведение сличений по шкалам Супер-Роквелла. Придание статуса для сличений on-going, в случае наличия свободных зон индентирования, после основного круга сличений, на мерах твердости

***Ключевые слова:** ключевые сличения, шкалы Роквелла, Государственный первичный эталон, меры твердости, область измерений, бюджет неопределенности, оценка неопределенности.*