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Final Report
On COOMET Vickers PTB/VNIIFTRI Key Comparison

(COOMET.M.H- K1.b and COOMET.M.H- K1.c)

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Abstract

This report describes a COOMET key comparison on Vickers hardness scales of two National Metrology Institutes - PTB and VNIIFTRI. The pilot laboratory was PTB, which was the linking institute with KCRV CCM.H-K1. In the key comparison two sets of hardness reference blocks for the Vickers hardness scales HV1 and HV30 consisting each of three hardness reference blocks with the hardness levels 240 HV, 540 HV and 840 HV are used.The same hardness reference blocks were used previously in CCM.H-К1 key comparisons. The measurement results and uncertainty assessments, announced by VNIIFTRI, are in good coordination with the key comparison reference values of CCM.H-К1.

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1 Introduction

The present key bilateral comparisons COOMET.M.H- K1.b and COOMET.M.H- K1.c were conducted between national metrology institutes of Germany (PTB) and Russia (VNIIFTRI). PTB declared readiness to act as the pilot laboratory of the comparison, which was the linking institute with KCRV CCM.H-K1.

2 Organisation

2.1 Participants

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2.2 Time schedule of the comparison

All of the PTB measurements presented in this report were those obtained at PTB during key comparison CCM.H-K1 in the period from March 2001 to March 2003. The following table shows the scheduled measuring time.

Institute/Country	Time of measurements
PTB, Germany	03/2001 - 03/2003
VNIIFTRI, Russia	04/2004

3 Standards

3.1 Description

In the key comparison two sets of hardness reference blocks for the Vickers hardness scales HV1 and HV30 consisting each of three hardness reference blocks with the hardness levels 240 HV, 540 HV and 840 HV (that is altogether six blocks) are used. The dimensions are as follows: length 60 mm, width 60 mm, thickness 10 mm. The upper side of the blocks which is the measurement surface is finished. The blocks are manufactured as commercial products by Buderus Co., Germany. For the comparison on the hardness reference blocks on the measurement surface a grid with 13 x 13 = 169 fields was engraved. At the left and the lower edge of the blocks numbers from 1 to 13 are engraved in order to define coordinates of the fields. The direction along the lower edge of the blocks defines the X-direction (lines), correspondingly the direction along the left edge of the blocks defines the Y-direction (rows) (see Fig. 1).

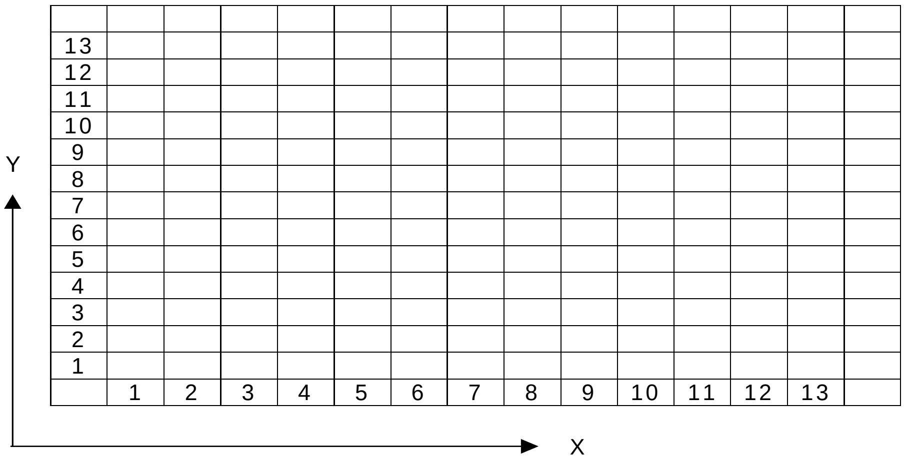

Fig. 1: Layout of the grid on the measurement surface of the hardness reference blocks

3.2 Handling

It is recommended to clean the blocks after unpacking with alcohol and then sign all fields reserved for your institute with a fiber pen in the left top corner. After measurement all dots on the blocks must be removed before packing in order to avoid corrosion.

4 Measurand

The measurands used in this comparison were of two kinds. Firstly, the mean value of each of the six hardness measurements on a hardness reference block had to be determined. The hardness measurements were made in the hardness scales HV1 and HV30 each for the nominal hardness levels 240 HV, 540 HV and 840 HV. The procedure of the hardness measurement is defined in ISO 6507-1 and -3. Secondly, the mean diagonal length of six reference indents had to be determined according to ISO 6507-1 and -3. The reference indents represent indents for the hardness levels 240 HV, 540 HV and 840 HV, each for the Vickers scales HV1 and HV30.
The same hardness reference blocks had been used previously in CCM.H-K1 key comparisons.

5 Methods of measurement

The methods of measurement and the measuring devices used by the participants are described in Appendix A1.

6 Measurement results

In the following tables the results for the hardness reference blocks with hardness levels of 240 HV, 540 HV and 840 HV are summarised. The results are expressed by the mean values of indentations in Table 1 and by evaluation of the reference indentation in Table 2.

Table 1: Mean values comparison (in HV)

Hardness scale	Value PTB	Unc. PTB	Value VNIIFTRI	Unc.VNIIFTRI	Diff.(VNIIFTRI-PTB)	Acceptable diff.
240 HV1	244,06	5,6	238,80	7,2	-5,26	9,12
240 HV30	238,00	1,5	238,52	2,3	0 ,52	2,75
540 HV1	539,03	11,6	534,50	18,2	-4,53	21,58
540 HV30	522,89	5,2	524,38	9,2	1,49	10,57
840 HV1	831,49	18,1	830,63	25,3	-0,86	31,11
840 HV30	817,86	9,9	818,13	17,1	0,27	19,76

The acceptable difference between the measurement result of PTB and VNIIFTRI follows from:

$$acceptable\_diff. \le \sqrt{(U_{PTB}^2 + U^2_{VNIIFTRI})}$$

The acceptable difference is confirmed by the stated uncertainties of the two participants of this bilateral comparison.

Table 2: Evaluation of the reference indentation(in µm)

Ref. Indents	Value PTB	St. Dev. PTB	Value VNIIFTRI	Std. Dev. VNIIFTRI	Diff.(VNIIFTRI-PTB)	Acceptable diff.
240 HV1	87,55	0,05	88,40	0,30	0,85	0,61
240 HV30	482,48	0,68	482,40	1,07	-0,08	2,54
540 HV1	58,58	0,12	58,35	0,16	-0,23	0,40
540 HV30	326,03	0,82	323,50	1,32	-2,53	3,11
840 HV1	47,38	0,11	47,15	0,19	-0,23	0,44
840 HV30	259,35	0,22	259,50	1,01	0,15	2,07

The acceptable difference between evaluation of the reference indentation is based on uncertainty approximations of optical systems suggested by PTB and VNIIFTRI.

$U_{L,PTB} = 2 * S_{L,PTB}$, $U_{L,VNIIFTRI} = 2 * S_{L,VNIIFTRI}$

$S_{L,PTB}$ - St. Dev. PTB; $S_{L,VNIIFTRI}$ - Std. Dev. VNIIFTRI.

$$acceptable\_diff. \le \sqrt{(U_{L,PTB}^2 + U^2_{L,VNIIFTRI})}$$

7 Uncertainty budgets

7.1 Calculation scheme

The calculation scheme can be seen from the example in Table 3.

Table 3: *Calculation scheme for the unified estimation of the uncertainty*

Influencing quantity X_i	Symbol	Unit	Value	Δx_i	s_i	a_i	$u^2(x_i)$	c_i	ΔH	$u^2(y_i)$	ν_i	$u_i^4(y)/\nu_i$
Test force F	***F***	N	**1,96**	0,000		9,80E-03	3,2E-05	1,3E+02	0,0E+00	5,1E-01	8	3,3E-02
Indentation diagonal length	***d***	mm	**0,039**	0		0,0004	5,3E-08	-1,3E+04	0,0E+00	8,7E+00	9	8,5E+00
Plane angle α	α	°	136	-8,7E-04		0,00175	1,0E-06	5,0E+01	-4,4E-02	2,5E-03	10	6,4E-07
Tip radius Δr	*r*	mm		3,0E-04		1,0E-04	3,3E-09	-7,1E+03	-2,1E+00	1,7E-01	10	2,8E-03
Length of line of junction Δc	*c*	mm		4,0E-04		1,0E-04	3,3E-09	9,0E+03	3,6E+00	2,7E-01	10	7,1E-03
Total									**1,41**	9,7E+00		8,5E+00
Combined standard uncertainty *u (H)*										3,1E+00	ν_{eff}	11
Confidence level										95%		
Coverage factor										2,2		
Expanded standard uncertainty *U (H)*										**6,8**	**HV**	
Expanded standard uncertainty *U(H)+I ΔHI*										**8,3**	**HV**	
Relative Expanded standard uncertainty $U_{rel}(H)$										**3,3**	**%**	
Hardness	**247,5**	**HV**										

From the influencing quantities X_i measurement deviations Δx_i and uncertainties in the form of standard deviation s_i (type A) and a_i (type B) are considered.
Standard uncertainty:

$$u^2(x_i) = s_i^2 \vee \frac{a_i^2}{3} \quad (1)$$

Sensitivity coefficients:

$$c_1 = \frac{\partial HV}{\partial F} = \frac{0{,}204 \cdot \sin\alpha / 2}{d^2}$$

$$c_2 = \frac{\partial HV}{\partial d} = \frac{-0{,}408 \cdot F \cdot \sin\alpha / 2}{d^3}$$

$$c_3 = \frac{\partial HV}{\partial \alpha} = \frac{0{,}102 \cdot F \cdot \cos\alpha / 2}{d^2} \quad (2)$$

$$c_4 = \frac{\partial HV}{\partial r} = \frac{-0{,}204 \cdot F \cdot \sin\alpha / 2}{d^3} \cdot (1{,}099 + 1{,}1515 \cdot \frac{r}{d})$$

$$c_5 = \frac{\partial HV}{\partial c} = \frac{0{,}2856 \cdot F \cdot \sin\alpha / 2}{d^3}$$

Single hardness deviation:

$$\Delta H_i = c_i \cdot \Delta x_i \quad (3)$$

Variances:

$$u^2(y_i) = c_i^2 u^2(x_i) \quad (4)$$

Combined standard uncertainty:

$$u(H) = \sqrt{\sum_{i=1}^{n} u^2(y_i)} \quad (5)$$

Sum of hardness deviations:

$$\Delta H = \sum_{i=1}^{n} \Delta H_i \quad (6)$$

Effective degrees of freedom, according to the Welch-Satterthwaite formula:

$$\nu_{eff} = \frac{u^4(y)}{\sum_{i=1}^{\nu} \frac{u_i^4(y)}{\nu_i}} \quad (7)$$

Coverage factor:

$$k = f(\nu_{eff}, P) \quad (8)$$

Expanded standard uncertainty:

$$U(H) = k \cdot u(H) + |\Delta H| \quad (9)$$

Relative expanded standard uncertainty:

$$U_{rel}(H) = \frac{U(H)}{H} \cdot 100, \% \quad (10)$$

According to this unified procedure for the estimation of measurement uncertainty, the following measurement uncertainties for the participants were received. If the participants had omitted the indenter tip radius because they did not have the facility to measure it, a value of r = 0,5 µm with an uncertainty of 0,2 µm was set in.

7.2 Results of calibrations

As basis for the calculation of the measurement uncertainty all participants carried out a calibration of the used Vickers standard machines. The results of the calibrations are summarised in the Table 4.

Table 4:
Results of the calibrations of Vickers standard machines used by the participants

			u_d, µm		u_α, °	r, µm	c, µm
	HV1	HV30	d<40µm	d>40µm			
VNIIFTRI	15,0 mN	0,1 N	0,4 µm	0,4-0,16 µm	0,02°	0,5 µm	0,5 µm
PTB	9,8 mN	0,15 N	0,3 µm	0,7-0,11 µm	0,1°	0,3 µm	0,4 µm

The data in Table 4 for force *F*, diagonal length *d* and indenter plane angle *α* indicate the calibration uncertainties, whereas for tip radius *r* and length of line of junction *c* the deviation are given.

7.3 Calculation of measurement uncertainty

As basis for the determination of the measurement uncertainty the draft guideline to the estimation of the uncertainty of the Brinell and the Vickers measuring method was recommended [2].
The uncertainty budgets of the participants based on the unified procedure as presented in ch. 7.1 appear in Appendix A2.

7.4 Data equivalence

There are data equivalence results for Vickers VNIIFTRI primary machine in attitude to KCRV CCM Vickers key comparison 2003 (KC CIPM).
Calculation of degree of equivalence performed at [3]. See in detail in Appendix A3.

Table 5.
Degree of equivalence of VNIIFTRI Vickers primary machine to KCRV CCM Vickers KC 2003 with respective uncertainties.

Hardness block	$d_{VNIIFTRI}$	$U(d_{VNIIFTRI})$
240 HV 1	-1,92	8,84
540 HV 1	-1,12	21,55
840 HV 1	3,52	40,98
240 HV 30	-1,54	4,38
540 HV 30	-2,40	12,70
840 HV 30	3,08	25,90

$d_{VNIIFTRI}$ – deviation of VNIIFTRI measurement results from KCRV of CCM Vickers KC 2003, $U(d_{VNIIFTRI})$ – uncertainties of this deviation.
Since in all cases abs $(\mathbf{d_{VNIIFTRI}}) < \mathbf{U}\ (\mathbf{d_{VNIIFTRI}})$, then measurement results and uncertainty assessments announced by VNIIFTRI are in good coordination with KCRV of CCM Vickers KC 2003 (CCM.H-K1).

8 Discussions, conclusions and remarks

The COOMET Vickers PTB/VNIIFTRI comparison can be considered as a successful metrological exercise. Representatively it delivered for two ranges of the test forces (small load Vickers and Macro-Vickers scales) valuable metrological data.

At present Vickers hardness reference blocks with high time-dependent stability and high local homogeneity, including high surface quality, are available.

It is recommended to concentrate metrological investigations on the following topics:

1). It was found that the calibration methods for the diagonal measurements, especially for diagonal lengths *d* > 100 µm, should be improved.

2). The calibration methods for the parameters of the indenter geometry, like tip radius and length of the line of junction, should be further developed.
3). For the further reduction of the uncertainty of Vickers measurements it is necessary to correct the indenter deviations and to provide indenters with higher quality.
4). For the diagonal measurements with optical microscopes the properties of the used optical system should be further investigated.

9. Acknowledgments

The authors thank A. Knott (NPL), who has significantly contributed to this comparison both by its analyzing and discussing the results, as well as critical reading of the manuscript.

10 References

[1] T. J. Quinn, Guidelines for key comparisons carried out by Consultative Committees, BIPM, Paris.
[2] EA Working group Hardness; Draft: Guideline to the estimation of the uncertainty of the Brinell and the Vickers measuring method, July 2002.
[3] Guidelines for data evaluation of COOMET key comparison. COOMET R/GM/14, 2006.

APPENDIX A1

DESCRIPTION OF INSTRUMENTS BY PARTICIPANTS

PTB

Instruments used to perform the indentations:

- Hardness standard machine VB 187,5 (combined for Vickers and Brinell scale, here used for HV 30 scale), dead weight machine for forces from 98 N until 1839 N with hydraulic drive of the deadweights.
  Manufactured by PGH Kraftmessgeräte Halle/Saale, electronical control for the hydraulic drive by PTB. The force application time and the duration time of the test force are set with hydraulic valves.
  Indenter by Carl Zeiss Jena Co.
- Hardness standard device HMV 2000, dead weight device, here used for the scale HV 1, manufactured by Shimadzu Co.

Instruments used to measure the indentations:

Scale	Device	Magnification	Numerical aperture
240 HV1	HMV2000	500	0,75
540 HV1	HMV2000	500	0,75
840 HV1	HMV2000	500	0,75
240 HV30	Libra200 (Leitz)	100	0,20
540 HV30	Libra200 (Leitz)	100	0,20
840 HV30	Libra200 (Leitz)	100	0,20

VNIIFTRI

The Russian national hardness standard on Vickers scales consists of a fixed device of direct loading of TPO-2 type (production of the plant «Etalon», S.-Petersburg) with a set of special weights which give the loading equal to 49.03; 98.07; 196.1; 294.2; 490.3 and 980.7 N, a microscope (production of the plant «LOMO», S.-Petersburg) with nominal division of scale equal to 0.8 µm with the numerical aperture of the objective 0.17 for the measurement of the indentation diagonal; a fixed device of direct loading of 2154 TBO (production of the plant «Tochpribor», Ivanovo) type with a set of special weights giving the loads of 9.807; 19.16; 49.03 and 98.07 N and a microscope (production of the plant «LOMO», S.-Petersburg) with nominal division of scale equal to 0,2 µm with the numerical aperture of the objective 0.65 for the measurement of the indentation diagonal; indenters (production of the plant «Tomal», Moscow region) which is a regular four-facet diamond pyramid with the angle between the opposite facets equal to 136° .

Instrument

Device type	Identifier
Russian National Hardness Standard for Vickers scales	GET 30-79

Tip. Indenter № 1162 (test force 9.807 N)

u_F	u_l	α (°)	r (µm)	c (µm)
0.01%	0.09%	0.02	0.3	0.5

Tip. Indenter № 1170 (test force 294.2 N)

u_F	u_l	α (°)	r (µm)	c (µm)
0.005 %	0.11%	0.02	0.5	0.5

APPENDIX A2

UNCERTAINTY BUDGETS OF PARTICIPANTS BASED ON UNIFIED PROCEDURE

PTB

240 HV 1

Influencing quantity *X_i*	**Symbol**	**Unit**	**Value**	***□x_i***	***s_i***	***a_i***	***u²(x_i)***	**c_i**	***□□***	***u²(y_i)***	***□_i***	***u_i⁴(y)/□_i***
Test force *F*	***F***	N	**9,81**	0,000		9,80E-03	3,2E-05	2,4E+01	0,0E+00	1,9E-02	8	4,6E-05
Indentation diagonal length	***d***	mm	**0,088**	0		0,0007	1,6E-07	-5,5E+03	0,0E+00	4,9E+00	9	2,6E+00
Plane angle *□*	*□*	°	136	-8,7E-04		0,00175	1,0E-06	4,8E+01	-4,2E-02	2,4E-03	10	5,7E-07
Tip radius *□r*	*r*	mm		3,0E-04		1,0E-04	3,3E-09	-3,0E+03	-9,0E-01	3,0E-02	10	9,1E-05
Length of line of junction *□c*	*c*	mm		4,0E-04		1,0E-04	3,3E-09	3,8E+03	1,5E+00	4,9E-02	10	2,4E-04
Total									**0,58**	5,0E+00		2,6E+00
Combined standard uncertainty *u (H)*										2,2E+00	□eff	9
Confidence level										95%		
Coverage factor										2,3		
Expanded standard uncertainty *U (H)*										**5,0**	**HV**	
Expanded standard uncertainty *U(H)+I □HI*										**5,6**	**HV**	
Relative Expanded standard uncertainty *U_rel (H)*										**2,3**	**%**	
Hardness	**244,1**	**HV**										

540 HV 1

Influencing quantity *X_i*	**Symbol**	**Unit**	**Value**	***x_i***	***s_i***	***a_i***	***u²(x_i)***	**c_i**	***Λ***	***u²(y_i)***	***i***	***u_i⁴(y)/ i***
Test force *F*	***F***	N	**9,81**	0,000		9,80E-03	3,2E-05	5,5E+01	0,0E+00	9,6E-02	8	1,1E-03
Indentation diagonal length	***d***	mm	**0,059**	0		0,0004	5,3E-08	-1,8E+04	0,0E+00	1,8E+01	9	3,5E+01
Plane angle		°	136	-8,7E-04		0,00175	1,0E-06	1,1E+02	-9,5E-02	1,2E-02	10	1,4E-05
Tip radius *r*	*r*	mm		3,0E-04		1,0E-04	3,3E-09	-1,0E+04	-3,0E+00	3,4E-01	10	1,1E-02
Length of line of junction	*c*	mm		4,0E-04		1,0E-04	3,3E-09	1,3E+04	5,1E+00	5,4E-01	10	3,0E-02
Total									**1,99**	1,9E+01		3,5E+01
Combined standard uncertainty *u (H)*										4,3E+00	eff	10
Confidence level										95%		
Coverage factor										2,2		
Expanded standard uncertainty *U (H)*										**9,6**	**HV**	
Expanded standard uncertainty *U(H)+I HI*										**11,6**	**HV**	
Relative Expanded standard uncertainty *U_rel (H)*										**2,2**	**%**	
Hardness	**539,0**	**HV**										

840 HV 1

Influencing quantity Xi	**Symbol**	**Unit**	**Value**	***ηx_i***	***s_i***	***a_i***	***u²(x_i)***	**c_i**	***η***	***u²(y_i)***	***η_i***	***u_i⁴(y)/η_i***
Test force *F*	***F***	N	**9,81**	0,000		9,80E-03	3,2E-05	8,5E+01	0,0E+00	2,3E-01	8	6,6E-03
Indentation diagonal length	***d***	mm	**0,047**	0		0,0003	3,0E-08	-3,5E+04	0,0E+00	3,7E+01	9	1,5E+02
Plane angle *η*	*η*	°	136	-8,7E-04		0,00175	1,0E-06	1,7E+02	-1,5E-01	2,9E-02	10	8,3E-05
Tip radius *ηr*	*r*	mm		3,0E-04		1,0E-04	3,3E-09	-2,0E+04	-5,9E+00	1,3E+00	10	1,6E-01
Length of line of junction *ηc*	*c*	mm		4,0E-04		1,0E-04	3,3E-09	2,5E+04	9,9E+00	2,0E+00	10	4,1E-01
Total									**3,88**	4,1E+01		1,6E+02
Combined standard uncertainty *u (H)*										6,4E+00	ηeff	10
Confidence level										95%		
Coverage factor										2,2		
Expanded standard uncertainty *U (H)*										**14,2**	**HV**	
Expanded standard uncertainty *U(H)+I ηHI*										**18,1**	**HV**	
Relative Expanded standard uncertainty *U_rel (H)*										**2,2**	**%**	
Hardness	**831,5**	**HV**										

240 HV 30

Influencing quantity X_i	Symbol	Unit	Value	x_i	s_i	a_i	$u^2(x_i)$	c_i	Λ	$u^2(y_i)$	$_i$	$u_i^4(y)/_i$
Test force *F*	***F***	N	**294,20**	0,000		1,50E-01	7,5E-03	8,2E-01	0,0E+00	5,0E-03	8	3,1E-06
Indentation diagonal length *d*	***d***	mm	**0,481**	0		0,0011	4,0E-07	-1,0E+03	0,0E+00	4,0E-01	9	1,8E-02
Plane angle		°	136	-8,7E-04		0,001745	1,0E-06	4,9E+01	-4,2E-02	2,4E-03	10	5,7E-07
Tip radius *r*	*r*	mm		4,0E-04		1,0E-04	3,3E-09	-5,5E+02	-2,2E-01	1,0E-03	10	1,0E-07
Length of line of junction *c*	*c*	mm		5,0E-04		1,0E-04	3,3E-09	7,0E+02	3,5E-01	1,6E-03	10	2,7E-07
Total									**0,09**	4,1E-01		1,8E-02
Combined standard uncertainty *u (H)*										6,4E-01	$_{eff}$	9
Confidence level										95%		
Coverage factor										2,3		
Expanded standard uncertainty *U (H)*										**1,5**	**HV**	
Expanded standard uncertainty *U(H)+I HI*										**1,5**	**HV**	
Relative Expanded standard uncertainty $U_{rel}(H)$										**0,6**	**%**	
Hardness	**238,0**	**HV**										

540 HV 30

Influencing quantity X_i	Symbol	Unit	Value	x_i	s_i	a_i	$u^2(x_i)$	c_i	Λ	$u^2(y_i)$	$_i$	$u_i^4(y)/_i$
Test force *F*	***F***	N	**294,20**	0,000		1,50E-01	7,5E-03	1,8E+00	0,0E+00	2,5E-02	8	8,0E-05
Indentation diagonal length	***d***	mm	**0,321**	0		0,0011	4,0E-07	-3,4E+03	0,0E+00	4,6E+00	9	2,3E+00
Plane angle		°	136	-8,7E-04		0,00175	1,0E-06	1,1E+02	-9,5E-02	1,2E-02	10	1,5E-05
Tip radius *r*	*r*	mm		4,0E-04		1,0E-04	3,3E-09	-1,9E+03	-7,4E-01	1,1E-02	10	1,3E-05
Length of line of junction	*c*	mm		5,0E-04		1,0E-04	3,3E-09	2,4E+03	1,2E+00	1,8E-02	10	3,4E-05
Total									**0,34**	4,6E+00		2,3E+00
Combined standard uncertainty *u (H)*										2,2E+00	$_{eff}$	9
Confidence level										95%		
Coverage factor										2,3		
Expanded standard uncertainty *U (H)*										**4,9**	**HV**	
Expanded standard uncertainty *U(H)+I HI*										**5,2**	**HV**	
Relative Expanded standard uncertainty $U_{rel}(H)$										**1,00**	**%**	
Hardness	**522,9**	**HV**										

840 HV 30

Influencing quantity X_i	Symbol	Unit	Value	□x_i	s_i	a_i	$u^2(x_i)$	c_i	□□	$u^2(y_i)$	□$_i$	$u_i^4(y)/$□$_i$
Test force *F*	***F***	N	**294,20**	0,000		1,50E-01	7,5E-03	2,8E+00	0,0E+00	5,9E-02	8	4,4E-04
Indentation diagonal length *d*	***d***	mm	**0,259**	0		0,0011	4,0E-07	-6,4E+03	0,0E+00	1,6E+01	9	3,0E+01
Plane angle □	□	°	136	-8,7E-04		0,00175	1,0E-06	1,7E+02	-1,5E-01	2,8E-02	10	8,1E-05
Tip radius □*r*	*r*	mm		4,0E-04		1,0E-04	3,3E-09	-3,5E+03	-1,4E+00	4,1E-02	10	1,7E-04
Length of line of junction □*c*	*c*	mm		5,0E-04		1,0E-04	3,3E-09	4,5E+03	2,2E+00	6,7E-02	10	4,4E-04
Total									**0,68**	1,7E+01		3,0E+01
Combined standard uncertainty *u (H)*										4,1E+00	□$_{eff}$	9
Confidence level										95%		
Coverage factor										2,3		
Expanded standard uncertainty *U (H)*										**9,2**	**HV**	
Expanded standard uncertainty *U(H)+I* □*HI*										**9,9**	**HV**	
Relative Expanded standard uncertainty $U_{rel}(H)$										**1,2**	**%**	
Hardness	**817,9**	**HV**										

VNIIFTRI

240 HV 1

Influencing quantity X_i	Symbol	Unit	Value	ηx_i	s_i	a_i	$u^2(x_i)$	c_i	η	$u^2(y_i)$	η_i	$u_i^4(y)/\eta_i$
Test force *F*	***F***	N	**9,81**	0,000		1,50E-02	7,5E-05	2,4E+01	0,0E+00	4,4E-02	8	2,5E-04
Indentation diagonal length	***d***	mm	**0,088**	0		0,0008	2,1E-07	5,5E+03	0,0E+00	6,5E+00	9	4,6E+00
Plane angle η	η	°	136	2,0E-02		0,01	3,3E-05	4,8E+01	9,7E-01	7,8E-02	10	6,0E-04
Tip radius ηr	*r*	mm		5,0E-04		1,0E-04	3,3E-09	-3,0E+03	-1,5E+00	3,0E-02	10	9,0E-05
Length of line of junction ηc	*c*	mm		5,0E-04		1,0E-04	3,3E-09	3,8E+03	1,9E+00	4,8E-02	10	2,3E-04
Total									**1,36**	6,7E+00		4,6E+00
Combined standard uncertainty *u (H)*										2,6E+00	η_{eff}	9
Confidence level										95%		
Coverage factor										2,3		
Expanded standard uncertainty *U (H)*										**5,8**	**HV**	
Expanded standard uncertainty *U(H)+I η HI*										**7,2**	**HV**	
Relative Expanded standard uncertainty $U_{rel}(H)$										**3,0**	**%**	
Hardness	**238,8**	**HV**										

540 HV 1

Influencing quantity X_i	Symbol	Unit	Value	ηx_i	s_i	a_i	$u^2(x_i)$	c_i	η	$u^2(y_i)$	η_i	$u_i^4(y)/\eta_i$
Test force *F*	***F***	N	**9,81**	0,000		1,50E-02	7,5E-05	5,5E+01	0,0E+00	2,2E-01	8	6,2E-03
Indentation diagonal length	***d***	mm	**0,059**	0		0,0006	1,2E-07	-1,8E+04	0,0E+00	4,0E+01	9	1,7E+02
Plane angle η	η	°	136	2,0E-02		0,01	3,3E-05	1,1E+02	2,2E+00	3,9E-01	10	1,5E-02
Tip radius ηr	*r*	mm		5,0E-04		2,0E-04	1,3E-08	-1,0E+04	-5,0E+00	1,4E+00	10	1,8E-01
Length of line of junction	*c*	mm		5,0E-04		2,0E-04	1,3E-08	1,3E+04	6,4E+00	2,2E+00	10	4,6E-01
Total									**3,48**	4,4E+01		1,7E+02
Combined standard uncertainty *u (H)*										6,6E+00	η_{eff}	10
Confidence level										95%		
Coverage factor										2,2		
Expanded standard uncertainty *U (H)*										**14,7**	**HV**	
Expanded standard uncertainty *U(H)+I η HI*										**18,2**	**HV**	
Relative Expanded standard uncertainty $U_{rel}(H)$										**3,4**	**%**	
Hardness	**534,5**	**HV**										

840 HV 1

Influencing quantity X_i	Symbol	Unit	Value	x_i	s_i	a_i	$u^2(x_i)$	c_i	Λ	$u^2(y_i)$	i	$u_i^4(y)/$ i
Test force *F*	***F***	N	**9,81**	0,000		1,50E-02	7,5E-05	8,5E+01	0,0E+00	5,4E-01	8	3,6E-02
Indentation diagonal length	***d***	mm	**0,047**	0		0,0004	5,3E-08	-3,5E+04	0,0E+00	6,6E+01	9	4,8E+02
Plane angle		°	136	2,0E-02		1,00E-02	3,3E-05	1,7E+02	3,4E+00	9,4E-01	10	8,8E-02
Tip radius *r*	*r*	mm		5,0E-04		2,0E-04	1,3E-08	-2,0E+04	-9,8E+00	5,1E+00	10	2,6E+00
Length of line of junction *c*	*c*	mm		5,0E-04		2,0E-04	1,3E-08	2,5E+04	1,2E+01	8,1E+00	10	6,5E+00
Total									**5,90**	8,1E+01		4,9E+02
Combined standard uncertainty *u (H)*										9,0E+00	eff	13
Confidence level										95%		
Coverage factor										2,2		
Expanded standard uncertainty *U (H)*										**19,4**	**HV**	
Expanded standard uncertainty *U(H)+I HI*										**25,3**	**HV**	
Relative Expanded standard uncertainty $U_{rel}(H)$										**3,0**	**%**	
Hardness	**830,6**	**HV**										

240 HV 30

Influencing quantity X_i	Symbol	Unit	Value	□x_i	s_i	a_i	$u^2(x_i)$	c_i	□>	$u^2(y_i)$	□$_i$	$u_i^4(y)$/□$_i$
Test force *F*	***F***	N	**294,20**	0,000		1,00E-01	3,3E-03	8,1E-01	0,0E+00	2,2E-03	8	6,0E-07
Indentation diagonal length *d*	***d***	mm	**0,483**	0		0,0015	7,5E-07	-9,9E+02	0,0E+00	7,3E-01	9	5,9E-02
Plane angle □	□	°	136	2.0E-02		0,01	3,3E-05	4,8E+01	1,9E-01	7,7E-02	10	6,0E-04
Tip radius □*r*	*r*	mm		5,0E-04		2,0E-04	1,3E-08	-5,4E+02	-2,7E-01	3,9E-03	10	1,5E-06
Length of line of junction □*c*	*c*	mm		5,0E-04		1,0E-04	3,3E-09	6,9E+02	3,5E-01	1,6E-03	10	2,5E-07
Total									**0,26**	8,2E-01		6,0E-02
Combined standard uncertainty *u (H)*										9,0E-01	□eff	11
Confidence level										95%		
Coverage factor										2,2		
Expanded standard uncertainty *U (H)*										**2,0**	**HV**	
Expanded standard uncertainty *U(H)+I* □*HI*										**2,3**	**HV**	
Relative Expanded standard uncertainty $U_{rel}(H)$										**0,9**	**%**	
238,5	**238,5**	**HV**										

540 HV 30

Influencing quantity X_i	Symbol	Unit	Value	□x_i	s_i	a_i	$u^2(x_i)$	c_i	□>	$u^2(y_i)$	□$_i$	$u_i^4(y)$/□$_i$
Test force *F*	***F***	N	**294,20**	0,000		1,00E-01	3,3E-03	1,8E+00	0,0E+00	1,1E-02	8	1,4E-05
Indentation diagonal length	***d***	mm	**0,326**	0		0,0016	8,5E-07	-3,2E+03	0,0E+00	8,8E+00	9	8,7E+00
Plane angle □	□	°	136	2,0E-02		1,00E-02	3,3E-05	1,1E+02	2,1E+00	3,7E-01	10	1,4E-02
Tip radius □*r*	*r*	mm		5,0E-04		1,0E-04	3,3E-09	-1,8E+03	-8,9E-01	1,0E-02	10	1,1E-05
Length of line of junction	*c*	mm		5,0E-04		1,0E-04	3,3E-09	2,3E+03	1,1E+00	1,7E-02	10	2,9E-05
Total									**2,36**	9,3E+00		8,7E+00
Combined standard uncertainty *u (H)*										3,0E+00	□eff	9
Confidence level										95%		
Coverage factor										2,3		
Expanded standard uncertainty *U (H)*										**6,9**	**HV**	
Expanded standard uncertainty *U(H)+I* □*HI*										**9,2**	**HV**	
Relative Expanded standard uncertainty $U_{rel}(H)$										**1,76**	**%**	
Hardness	**524,4**	**HV**										

840 HV 30

Influencing quantity X_i	Symbol	Unit	Value	□x_i	s_i	a_i	$u^2(x_i)$	c_i	□>	$u^2(y_i)$	□$_i$
Test force *F*	***F***	N	**294,20**	0,000		1,00E-01	3,3E-03	2,8E+00	0,0E+00	2,6E-02	8
Indentation diagonal length *d*	***d***	mm	**0,261**	0		0,0016	8,5E-07	-6,3E+03	0,0E+00	3,4E+01	9
Plane angle □	□	°	136	2,0E-02		1,00E-02	3,3E-05	1,7E+02	3,3E+00	9,1E-01	10
Tip radius □*r*	*r*	mm		5,0E-04		2,0E-04	1,3E-08	-3,5E+03	-1,7E+00	1,6E-01	10
Length of line of junction □*c*	*c*	mm		5,0E-04		1,0E-04	3,3E-09	4,4E+03	2,2E+00	6,4E-02	10
Total									**3,78**	3,5E+01	
Combined standard uncertainty *u (H)*										5,9E+00	□eff
Confidence level										95%	
Coverage factor										2,3	
Expanded standard uncertainty *U (H)*										**13,3**	**HV**
Expanded standard uncertainty *U(H)+I* □*HI*										**17,1**	**HV**
Relative Expanded standard uncertainty $U_{rel}(H)$										**2,1**	**%**
Hardness	**818,1**	**HV**									

APPENDIX A3

CALCULATION OF VNIIFTRI DEGREE OF EQUIVALENCE (DoE) RELATIVELY TO KCRV CCM Vickers KC.

PTB hardness laboratory was the pilot laboratory of CCM Vickers key comparison 2003. The hardness scales HV1 and HV30 comparison were the objects of these comparison.

RMO KC COOMET Vickers PTB/VNIIFTRI comparison used the same hardness blocks (for scales HV1 and HV30), which were used in KC CIPM.

Calculation of degree of equivalence performed at "COOMET Guide to assessment data of KC" (COOMET R/GM/14:2006).

Parameters of connecting NMI (PTB) Procedure C , ch. 5.1.1. COOMET R/GM/14:2006 used for calculation				
Hardness block	x^*	x''^*	Δ-additive adjustment	$u^2(\Delta)$
240 HV 1	243,15	244,06	-0,91	9,26
540 HV 1	537,76	539,03	-1,27	39,44
840 HV 1	835,79	831,49	4,3	81,52
240 HV 30	-	238,0	-0,91**	0,63
540 HV 30	-	522,89	-1,27**	12,5
840 HV 30	-	817,86	4,3**	22,98

$\Delta = x^* - x''^*$;
x^*- PTB results in CCM Vickers KC (march 2001)
x''^*- PTB results in CCM Vickers KC (march 2003) (results reported by PTB in the RMO Vickers KC)
$u^2(\Delta)=S^2_{2001}+ S^2_{2003}$; (for HV 1) S_{2001} - Std. Dev. of measurement results reported in CCM Vickers KC (march 2001); S_{2003} - Std. Dev. of measurement results reported in CCM Vickers KC (march 2003);
$u^2(\Delta)=2S^2_{2003}$; (for HV 30); S_{2003} - Std. Dev. of measurement results reported in CCM Vickers KC (march 2003);

** Since in RMO KC COOMET Vickers PTB/VNIIFTRI comparison there are no data on the test blocks drift in 2004, it was decided to use the value of the test blocks drift measured from 2001 to 2003 during CCM. H-K1 key comparisons as the estimate of the hardness test blocks drift. The pilot of CCM H-K1 key comparisons measured the test blocks drift on the HV 1 scale and didn't measure it on the HV 30 scale, that's why it was decided to consider the test blocks drift on the HV1 scale from CCM. H-K1 key comparisons as the upper level of the test blocks drift during comparisons on HV1 and HV30 scales.

Calculation of degree of equivalence for VNIIFTRI Vickers primary machine Procedure C , ch. 5.2. COOMET R/GM/14:2006 used for calculation					
Hardness block	x_{ref}	$X_{VNIIFTRI}$	dif	Δ	**$d_{VNIIFTRI}$**
240 HV 1	239,81	238,8	-1,01	-0,91	**-1,92**
540 HV 1	534,35	534,5	0,15	-1,27	**-1,12**
840 HV 1	831,41	830,63	-0,78	4,3	**3,52**
240 HV 30	239,15	238,52	-0,63	-0,91	**-1,54**
540 HV 30	525,51	524,38	-1,13	-1,27	**-2,40**
840 HV 30	819,35	818,13	-1,22	4,3	**3,08**

x_{ref} – KCRV of CCM Vickers KC

$x_{VNIIFTRI}$ – VNIIFTRI measurement results values obtained in RMO KC

dif= $x_{VNIIFTRI}$ - x_{ref}

$d_{VNIIFTRI}$= $x_{VNIIFTRI}$ +Δ-x_{ref}

Uncertainty of VNIIFTRI measurements results about KC CIPM Procedure C , п.5.2. COOMET R/GM/14:2006 used for calculation					
Hardness block	$u^2(x_{VNIIFTRI})$	$u(x_{ref})$ from KCDB	$u^2(x_{ref})$	$u^2(\Delta)$	**$u^2(d_{VNIIFTRI})$**
240 HV 1	6,7	2,08	4,33	9,26	**19,55**
540 HV 1	44,2	6,1	37,21	39,44	**116,12**
840 HV 1	80,7	16,82	282,91	81,52	**419,86**
240 HV 30	0,82	1,97	3,88	0,63	**4,79**
540 HV 30	9,3	5,03	25,30	12,5	**40,35**
840 HV 30	35,0	11,25	126,56	22,98	**167,76**

$u^2(x_{VNIIFTRI})$ – VNIIFTRI uncertainties announced in RMO KC (see in Appendix A2).

$u^2(x_{ref})$ – uncertainties of KCRV of CCM Vickers KC

$u^2(\Delta)$ – uncertainties of additive adjustment of connecting NIM (PTB).

$u^2(x_{PTB})$ – uncertainties of PTB in CCM Vickers KC

$$u^2(d_{VNIIFTRI}) = u^2(x_{VNIIFTR}) + u^2(x_{ref}) + u^2(\Delta)\left\langle 1 - u^2(x_{ref}) / u^2(x_{PTB})\right\rangle$$

Acknowledgement of uncertainties announced			
Hardness block	abs(**$d_{VNIIFTRI}$**)	2u (**$d_{VNIIFTRI}$**)	Condition of acknowledgement is executed
240 HV 1	1,91	8,84	Yes
540 HV 1	1,12	21,55	Yes
840 HV 1	3,52	40,98	Yes
240 HV 30	1,54	4,38	Yes
540 HV 30	2,40	12,70	Yes
840 HV 30	3,08	25,90	Yes

Since in all cases abs($\mathbf{d}_{VNIIFTRI}$) < 2u($\mathbf{d}_{VNIIFTRI}$), the results of the uncertainty assessments announced by VNIIFTRI are in good coordination with KCRV of CCM Vickers KC.

Degree of equivalence of VNIIFTRI Vickers primary machine to KCRV KC CIPM with respective uncertainties.		
Hardness block	$\mathbf{d}_{VNIIFTRI}$	U($\mathbf{d}_{VNIIFTRI}$)
240 HV 1	**-1,91**	8,84
540 HV 1	**-1,12**	21,55
840 HV 1	**3,52**	40,98
240 HV 30	**-1,54**	4,38
540 HV 30	**-2,4**	12,70
840 HV 30	**3,08**	25,90