

REPORT

CCM Key Comparison of Vickers Hardness Scales

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Abstract

This report details the work carried out as part of a key comparison of the Vickers hardness scale, between the hardness laboratories of national metrology institutes (NMIs). The Physikalisch-Technische Bundesanstalt (PTB, Braunschweig, Germany) acted as the pilot laboratory for the comparison.

The Key Comparison covered the Vickers hardness scales HV 0,2, HV 1, and HV 30, although only the HV 30 work was done due to the other scales not being supported.

The results obtained were sent back to PTB for comparison with the other NMIs.

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Approved on behalf of Managing Director, NPL,
by Dr R Preston, Authorised by Head of NPL Material Centre

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1. Introduction

During the 2nd meeting of the Ad hoc Working Group on Hardness on May 10th, 1999 in the BIPM in Paris, it was agreed to carry out a key comparison of Vickers hardness scales in which the hardness laboratories of national metrology institutes (NMIs) should participate. The Physikalisch-Technische Bundesanstalt (PTB, Braunschweig, Germany) acted as the pilot laboratory for the comparison.

2. Description of the Key Comparison

In the key comparison nine hardness blocks were used, three Vickers hardness scales (HV 0,2, HV 1 and HV 30) were covered, each consisting of three hardness reference blocks with nominal hardness values of 240 HV, 540 HV and 840 HV. Only the HV 30 scale was measured at NPL, as it is the only scale currently supported by the NPL hardness measurement service.

The blocks had dimensions of 60 mm by 60 mm, with a thickness of 10 mm, and were manufactured by Buderus Co. Germany.

For the comparison, the hardness reference blocks were engraved with a 13 by 13 grid, giving 169 fields. The grids were labeled with engraved numbers from 1 to 13 in order to define the coordinates of the fields, (see Figure 1).

The hardness reference blocks were sent to each participant in accordance with the measurement timetable, (Table 1).

The pilot laboratory made measurements at the beginning and end of the key comparison in order to evaluate the stability of the hardness reference blocks.

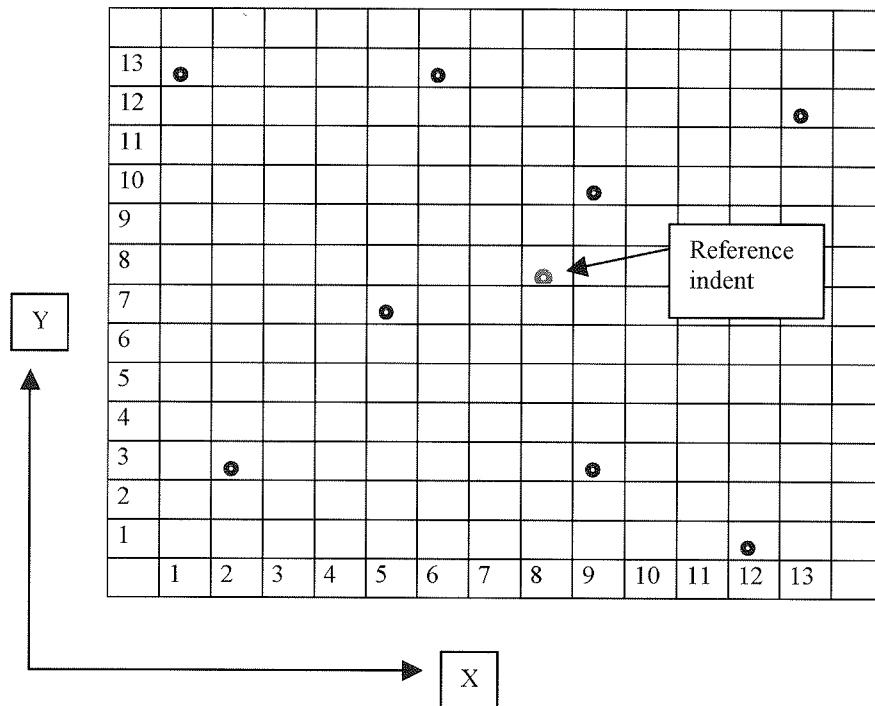


Figure 1. Layout of the grid on the measurement surface of the hardness reference blocks and locations for NPL indents and reference indent.

3. Procedure

Before the measurements, each participant carried out a calibration of their hardness machine, including the test force, the length measuring system, and the indenter. Results of the calibration are the uncertainties of:

- 1) The test force (u_F),
- 2) The length measuring system (u_l),
- 3) The measurement results of the indenter, i.e. the plane angle α and length of the line of junction c .

The blocks were cleaned with alcohol after unpacking and then all fields reserved for NPL were marked with a fibre pen on the top left corner. After the measurements all dots on the blocks were removed with alcohol, to avoid corrosion.

On each block in rows 7 and 8, each laboratory made one initial indentation, which was not measured. This was to help settle the block before the measured readings.

Each participant then made eight indentations on each hardness reference block and measured the diagonal lengths of the indentations according to the procedure described in ISO 6507-3 [1]. The number of indentations allows evaluation of the stochastic deviations occurring during the measurements as well as the inhomogeneity of the hardness distribution on the

hardness reference blocks. The horizontal and vertical diagonal lengths of the indentations were measured three times each. The average value of the horizontal and vertical lengths were then used to calculate the overall Vickers hardness.

The locations of the eight indentations on each block for NPL are displayed in Figure 1. Each participating institute was informed only of the coordinates for their own indentations, in order to guarantee the anonymity of each participant.

The nearest free place in the rows 7 and 8 was used in order to repeat any measurement needing to be repeated.

Each participant also measured the diagonal lengths of one reference indentation on each hardness reference block. This was to make it possible to separate the influence of the measurement uncertainty from the overall uncertainty of the Vickers measurement. The coordinates of the reference indentations were (8,8) for each block.

Each participating institute in this key comparison made and measured a maximum of 72 indentations and measured 9 reference indentations. NPL for example only made and measured indentations on 3 blocks, within the HV 30 scale.

The nine hardness reference blocks were stored in a wooden box for transportation. The receipt and the sending of the hardness reference blocks was reported to the next participant and to the pilot laboratory with the fax form in Annex A. For packing, the blocks were cleaned with alcohol, then wrapped in anti-rusting paper, put in a plastic bag and placed in the wooden box. Finally, the wooden box was closed with screws.

4. Timetable of the measurements (preliminary)

Table 1 shows the preliminary timetable for when each lab was to make their measurements. The dates were only preliminary and during the course of the comparison there were several delays at different institutes, causing the dates to be changed, so they are not accurate. The order the institutes made their measurements remained the same.

Table 1. Timetable of the measurements (preliminary).

Institute/Country	Time of measurements
PTB, Germany	02/2001
IMGC, Italy	03/2001
INMETRO, Brazil	05/2001
PTB, Germany	07/2001
NIST, USA	08/2001
NRLM, Japan	09/2001
NIM, PR China	10/2001
KRISS, Republic of Korea	11/2001
CMI, Czech Republic	12/2001
GUM, Poland	01/2002
NPL, UK	02/2002
PTB, Germany	03/2002

5. Data report

5.1 Equipment

The work was carried out using NPL's 1.5 kN hardness machine (shown in Figure 2), which applies forces from 30 N to 1.5 kN.



Figure 2. NPL 1.5 kN Hardness Machine.

The machine is designed with twin reaction columns to eliminate torque from the screw drive. High accuracy load cells, traceable to NPL's force standard machines, measure the applied force. Indenter depth measurement is by a laser interferometer system. This interferometer is traceable to the UK realisation of the metre at NPL. Time control of the loading cycle and measurement of the indenter velocity is calibrated against the UK time standard at NPL.

The 1.5 kN hardness machine is based on the Instron RT2000 hardness machine, and is able to cover demands of the Rockwell scales HRA-HRK and superficial Rockwell scales from HR15N to HR45T [45]. In addition, the machine is able to meet the forces requirements of Vickers scales from HV5 to HV100 and the Brinell scales from HB2/4 to HB5/125. The machine is interfaced to its own PC-driven control system based upon Instron's Merlin and Wavemaker software product. This software uses generalised waveforms for closed loop control. The servo design provides control of the applied force and indenter motion and avoids the risk of load overshoot present in some deadweight machines.

5.2 Vickers indentation measuring device

The Vickers indentation measurement system consists of a Reichert-Jung (Polyvar Met) microscope with CCD camera to image the indentation, an interferometer to measure the movement of the stage, and image analysis software that controls the measurement process, Figure 3. The software system has been developed by Graftek Italia Srl for IMGC. This system minimises human manipulation of “cross hairs” in identifying the diagonal vertices of a Vickers indentation in an automated process.



Figure 3. Indentation measuring system.

5.3 Magnifications and numerical apertures of the lenses used

A range of lenses were used for measuring the indentations the details of these are given in Table 2.

Table 2. Lenses used for the measurements of the different indentations.

HV30 Scale	Hardness	Primary lens		Secondary lens (Not including X10 internal magnification)
		Magnification	N/A	
	240	X10	0.25	X0.8
	540	X10	0.25	X1.25
	840	X20	0.5	X0.8

5.4 Results of calibration

Before the comparison work was started, the equipment used was re-calibrated to provide a more accurate uncertainty evaluation, as was required for this CCM comparison.

5.4.1 Test force

The force calibration included the nominal test force of 300 N to be calibrated against the NPL standard deadweight machine via a reference load cell. This force is the relevant force for the Vickers HV 30 scale.

The calibration shows the machine operates with an error of +0.007 % at the 300 N force. There was also a 0.03 % uncertainty in the stability of force of this force.

5.4.2 Length measuring system

The laser was calibrated, and comparative results of direct measurements of an indentation diameter, using only the laser to measure the indentation, and indirect measurements of the same indentation via the screen pixels (calibrated against the laser), were made:

Comparative results:

240 HV 30:	Direct:	(h) 481.517 μm ,	(v) 482.353 μm
	Indirect:	(h) 481.693 μm ,	(v) 482.340 μm
540 HV30:	Direct:	(h) 325.410 μm ,	(v) 326.190 μm
	Indirect:	(h) 325.465 μm ,	(v) 326.137 μm
840 HV 30:	Direct:	(h) 257.330 μm ,	(v) 257.610 μm
	Indirect:	(h) 257.330 μm ,	(v) 257.770 μm

These results show the difference between the direct and indirect methods and show a variation of less than 0.2 μm in both the X and Y direction. Anything less than 0.5 μm is believed to be reasonable and although included in the uncertainty budget, it does not cause any significant uncertainty contribution to the overall results.

The actual measurements were made using the indirect method.

5.4.3 Parameters of indenter

The indenter used for this comparison was a Euro Products Vickers indenter with the following geometric characteristics:

Plane angle: 136.143°

Length of line of junction: 0.37 μm

5.5 Uncertainty evaluation

Table 3. Uncertainty evaluation

Quantity	Value	Divisor	Standard uncertainty	Source of uncertainty	Uncertainty in hardness, $u(x_i)$.Ci		
x_i			$U(x_i)$		840	540	240
u_F (N)	0.294	2	0.147	Test force	0.420	0.270	0.120
u_l [%]	0.14	2	0.07	Diagonal length	-1.177	-0.739	-0.336
α [°]	0.1	$\sqrt{3}$	0.0577	Plane angle	0.171	0.110	0.049
Δc [mm]	0.001	$\sqrt{3}$	0.000577	Line of junction	-0.057	-0.004	-0.002
Standard uncertainty / HV					1.26	0.79	0.36
Expanded uncertainty / HV, $k=2$					2.53	1.59	0.72

Table 3 shows the uncertainty evaluation for the measurements within this comparison. It is separated into the three hardness values of the blocks used, and the uncertainties that can be quoted with the measurements made. All the uncertainty values were calculated from the data obtained during the recent calibration, as well as historical data, of the hardness machine, indenter and indentation measuring system.

These uncertainties are calculated using the sensitivity coefficients for the HV 30 scale, which are based on the Vickers equation (1) given below:

$$HV = 0,102 \times 2F \sin\left(\frac{\alpha}{2}\right) / d^2 \quad (1)$$

Where:

F = force (in N)

α = plane angle of the indenter (136°)

d = mean indentation diagonal length (in mm)

Partial derivatives allow the sensitivity coefficients for force, indenter angle, and indentation diagonal length to be determined from this.

$$\frac{\partial HV}{\partial F} = \frac{HV}{F}$$

$$\frac{\partial HV}{\partial \alpha} = \frac{HV}{2 \tan\left(\frac{\alpha}{2}\right)}$$

$$\frac{\partial HV}{\partial d} = -2 \times \frac{HV}{d}$$

Also geometrical considerations allow the sensitivity coefficients for length of line of junction to be approximated:

$$\frac{\partial HV}{\partial c} = -1.5 \left(\frac{c}{d}\right)^2$$

Where:

c = length of line of junction (in mm)

The assumption made is that the volume of the indentation remains the same, for varying values of c , as would be the case with an indenter of perfect geometry.

6. Results:

Table 4 shows the measured results of the reference indentations on each of the three HV30 blocks.

Tables 5, 6 and 7 show the measured results of the indentations made by NPL on the 240, 540 and 840 HV 30 blocks respectively. The horizontal and vertical measurements are listed with their means and subsequent calculated hardness.

Table 4. Reference Indentation Measurements

Reference block	Hardness (HV)	d _{h1} (μm)	d _{h1} (μm)	d _{h1} (μm)	d _{v1} (μm)	d _{v1} (μm)	d _{v1} (μm)	d _{hmean} (μm)	d _{vmean} (μm)	d (μm)	Temp (°C)
240	30	485.97	485.99	485.97	482.73	482.76	482.8	485.98	482.76	484.37	21.2°-21.8°
540	30	327.08	327.06	327.08	324.66	324.66	324.65	327.07	324.66	325.865	21.1°-21.6°
840	30	259.01	259.06	259.04	256.53	256.55	256.52	259.04	256.53	257.785	21.3°-21.8°

Table 5. 240 HV 30 block results

Test identifier	No. of test	hardness scale	d _{h1} (μm)	d _{h2} (μm)	d _{h3} (μm)	d _{v1} (μm)	d _{v2} (μm)	d _{v3} (μm)	d _{hmean} (μm)	d _{vmean} (μm)	d (μm)	Hardness (HV 30)	Temp (°C)
	1	HV 30	481.69	481.7	481.69	482.33	482.3	482.39	481.69	482.34	482.02	239.51	21.2° To 21.8°
	2		482.64	482.64	482.65	482.72	482.69	482.7	482.64	482.70	482.67	238.85	
	3		483.12	483.12	483.09	483.89	483.76	483.86	483.11	483.84	483.47	238.06	
	4		482.8	482.74	482.74	483.23	483.17	483.2	482.76	483.20	482.98	238.55	
	5		483.11	483.17	483.17	483.57	483.43	483.48	483.15	483.49	483.32	238.21	
	6		483.56	483.46	483.51	483.91	483.89	483.92	483.51	483.91	483.71	237.83	
	7		482.46	482.45	482.39	483.51	483.47	483.46	482.43	483.48	482.96	238.57	
	8		482.44	482.43	482.56	482.9	482.91	482.87	482.48	482.89	482.69	238.84	

Table 6. 540 HV 30 block results

Test identifier	No. of test	hardness scale	d _{h1} (μm)	d _{h2} (μm)	d _{h3} (μm)	d _{v1} (μm)	d _{v2} (μm)	d _{v3} (μm)	d _{hmean} (μm)	d _{vmean} (μm)	d (μm)	Hardness (HV 30)	Temp (°C)
	1	HV 30	325.63	325.46	325.47	326.23	326.08	326.1	325.52	326.14	325.83	524.16	21.1°
	2		325.19	325.16	325.22	325.3	325.35	325.29	325.19	325.31	325.25	526.02	
	3		325.14	325.17	325.11	325.17	325.19	325.19	325.14	325.18	325.16	526.31	To
	4		325.86	325.87	325.88	326.29	326.29	326.3	325.87	326.29	326.08	523.34	21.6°
	5		325.53	325.46	325.48	326.01	326.09	326.08	325.49	326.06	325.78	524.33	
	6		325.37	325.37	325.32	326.28	326.32	326.29	325.35	326.30	325.83	524.17	
	7		325.46	325.48	325.42	325.84	325.83	325.82	325.45	325.83	325.64	524.76	
	8		325.08	325.09	325.13	325.63	325.55	325.44	325.10	325.54	325.32	525.80	

Table 7. 840 HV 30 block results

Test identifier	No. of test	hardness scale	d _{h1} (μm)	d _{h2} (μm)	d _{h3} (μm)	d _{v1} (μm)	d _{v2} (μm)	d _{v3} (μm)	d _{hmean} (μm)	d _{vmean} (μm)	d (μm)	Hardness (HV 30)	Temp (°C)
	1	HV 30	257.37	257.35	257.32	257.77	257.77	257.77	257.35	257.77	257.56	838.86	21.3°
	2		257.97	257.96	257.96	258.31	258.36	258.29	257.96	258.32	258.14	835.07	
	* 3		257.81	257.88	257.83	257.99	257.99	257.91	257.84	257.96	257.90	836.62	To
	4		257.29	257.25	257.22	257.49	257.53	257.61	257.25	257.54	257.40	839.90	21.8°
	5		258.12	258.07	258.05	257.74	257.81	257.8	258.08	257.78	257.93	836.43	
	6		257.78	257.78	257.74	258.16	258.17	258.15	257.77	258.16	257.96	836.22	
	7		257.58	257.57	257.56	257.94	257.99	257.93	257.57	257.95	257.76	837.53	
	8		258.02	258.01	258.02	257.76	257.71	257.76	258.02	257.74	257.88	836.76	

* For the 840 HV 30 block, the location of the third indentation was changed to (4,7) because there was already an indent in the required location (5,7).

Table 8. Expanded uncertainties of the mean values.

NPL Measurements [HV 30]									Expanded uncertainty of the single meas.	Standard deviation of the meas.	Mean value	Expanded uncertainty of the mean value
	1	2	3	4	5	6	7	8	[HV]	[HV]	[HV]	[HV]
HV30												
240	239.51	238.85	238.06	238.55	238.21	237.83	238.57	238.84	0.72	0.53	238.55	0.81
540	524.16	526.02	526.31	523.34	524.33	524.17	524.76	525.8	1.59	1.06	524.86	1.76
840	838.86	835.07	836.62	839.9	836.43	836.22	837.53	836.76	2.53	1.55	837.17	2.76

Table 8 shows the mean hardness values measured for each indentation for the three hardness blocks, including their standard deviation and the expanded uncertainty of the mean value.

The combined standard uncertainty of the mean value is based on the square root of the sum of the squares, of the standard uncertainty of the single measure, (half the expanded uncertainty), and the standard deviation over the square root of the number of measurements.

7. Conclusions

Although no real conclusions can be made from the work within this comparison, due to the results from NPL being the only results available, it is still possible to look at certain aspects of the work.

Looking at Table 8, it can be seen that the standard deviation of the measurements are reasonable. The greatest being 1.55 HV for the 840 HV 30 block. All the standard deviations are less than the uncertainties of the measurements, for their respective blocks, showing that all the results are as would be expected and that there are no notably poor measurements. Looking at the measurements for the different blocks, tables 5-7, it can be seen that there is good repeatability in the measurements of the indentations, with an average standard deviation of 0.03 in both the horizontal and vertical directions, showing no notable problem with the indentation measurement system.

Once the results from all the NMIs are processed by PTB, and results distributed, further conclusions will be possible.

8. Acknowledgements

The author of this report acknowledges the financial support of the National Measurement System Policy Unit of the UK Department of Trade and Industry.

9. References

- [1] ISO 6507-3: Metallic materials - Vickers hardness test - Calibration of reference blocks
- [2] GUM: Guide to the Expression of Uncertainty in Measurement; 1. edition 1993, corrected and reprinted 1995, International Organization for Standardization, Geneva, Switzerland
- [3] Draft of EA working group "Hardness": Guideline to the determination of the uncertainty of the Brinell and the Vickers measuring method, 09/1999

10. Annexes

10.1 Annex A

Fax form:

Notification of receipt and sending

To:	Physikalisch-Technische Bundesanstalt Dr. Konrad Herrmann Bundesallee 100 38116 Braunschweig Germany
Fax:	+49 531 592 5105

From:	National Physical Laboratory Force Standards Section Centre for Mechanical and Acoustical Metrology Mr. Laurence Brice Queens Road. Teddington. Middlesex TW11 0LW. United Kingdom
Fax:	+44 (0)20 89436184
Date:	

Re: CCM-WGH Key Comparison
Vickers hardness scales
Notification of receipt/sending

I have received on date _____ the nine Vickers hardness reference blocks in good/bad condition.

I have sent off on date _____ the nine Vickers hardness reference blocks to the next participant in the key comparison:

Address:

Physikalisch-Technische Bundesanstalt
Dr. Konrad Herrmann
Bundesallee 100
38116 Braunschweig
Germany

Notes:

Signature:

10.2 Annex B

Addresses of the participating institutes

Institute	Address	Tel/Fax/e-mail
PTB	Physikalisch-Technische Bundesanstalt Leiter des FL 5.12, Herrn Dr. K. Herrmann Bundesallee 100 38116 Braunschweig Germany	Tel: +49 531 592 5140 Fax: +49 531 592 5105 konrad.herrmann@ptb.de
IMGC	Istituto di Metrologia "G. Colonnetti" Dr. A. Germak Strada delle cacce, 73 10135 Torino Italy	Tel: +39 011 3977 367 Fax: +39 011 3977 426 A.Germak@imgc.to.cnr.it
NPL	National Physical Laboratory Force Standards Section Centre for Mechanical and Acoustical Metrology Mr. Laurence Brice Queens Road Teddington, Middlesex United Kingdom TW11 0LW	Tel: +44 20 8943 7054 Fax: +44 20 8943 6184 Laurence.Brice@npl.co.uk
INMETRO	National Institute of Metrology, Standardization and Industrial Quality Force & Hardness Laboratory Mr. Renato Reis Machado Av. N. S. das Gracias, 50 - Xerem 25250-020, Duque de Caxias, RJ, Brasil	Tel: +5521 679-1311 Fax: +5521 679 1420 rrmachado@innet.inmetro.gov.br
NIST	National Institute of Standards and Technology Materials Performance Group Metallurgy Division Materials science and Engineering Laboratory Mr. Samuel R. Low 100 Bureau drive, Mail Stop 8553 Gaithersburg, Maryland 20899 U.S.A.	Tel: 001 301 975 5709 Fax: 001 301 975 4553 samuel.low@nist.gov
NRLM	National Research Laboratory of Metrology Instrumentation Mathematics Section Mr. Hajime Ishida 1-1-4 Umezono Tsukuba, Ibaraki 305 Japan	Tel: +81 298 61 4043 Fax: +81 298 61 4061 ishida@nrlm.go.jp
NIM	National Institute of Metrology Mechanics Division Director of Hardness Laboratory Mr. He Li 18, Bei San Huan Dong Lu 100013 Beijing P. R. China	Tel: +86 1 6421 8628 Fax: +86 1 6421 8703 Liqznim@hotmail.com

KRISS	Korea Research Institute of Standards and Science Microstructure Analysis Group Materials Evaluation Center Mr. Gun-Woong Bahng P.O.Box 102 Yusong, Taejon 305 - 600 Republic of Korea	Tel: +82 42 868 5320 Fax: +82 42 868 5032 gwbahng@kriss.re.kr
CMI	Cesky Metrologický Institut Laboratoire primární metrologie Mr. Jiri Borovsky V botanice 4 15072 Praha Czech Republic	Tel: +420 2 5732 5579 Fax: +420 2 5732 8077 jborovsky@cmi.cz
GUM	Główny Urząd Miar Zakład Masy i Siły Mrs. A. Osinska-Karczmarek ul. Elektoralna 2 00-950 Warszawa Poland	Tel: +22 6 200 241 Fax: +22 6 208 378 gum@gum.gov.pl