

# Study for the requalification of Inmetro's Primary Hardness Standardization Machine for Vickers HV3 scale

J T Filho<sup>1,2</sup>, R Q Fratarì<sup>2</sup>, C R S Azeredo<sup>2</sup> and S P Oliveira<sup>2</sup>

<sup>2</sup> Force Laboratory, National Institute of Metrology Quality and Technology, Av. Nossa Senhora das Gracas, 50, Predio 3, Duque de Caxias, RJ 25250-020, Brazil

<sup>1</sup> E-mail: jtfilho@inmetro.gov.br

**Abstract.** The standardization of Vickers hardness quantity in Brazil was established as related to the quality management system and the properties of metallic materials that are used in industries like steelworks, car makers aircraft manufacturers. This work aimed in describing the metrological methodology applied in the requalification of the Inmetro's Primary Hardness Standardization Machine for low, medium and high hardness ranges of Vickers HV3 scale.

## 1. Introduction

Hardness is the most used mechanical property and has widespread recognition due to its essential importance in both production and service performance of many items produced in virtually all industries, the most important being the metal-mechanical, steel, metallurgy, automobile, aerospace, tools and machinery and equipment sectors. There is no unique definition for hardness. It can be defined as the resistance to permanent plastic deformation, to absorbed energy during the impact, and to the risk or penetration of a harder material in a softer one. Industrially, the last approach is the one used for metals.

Hardness measurements are run several times in the production line in order to assure the quality of various items produced by sampling some parts from time to time along the production lines and in raw-materials receiving tests. It is the most evaluated property during the research and development (R&D) activities made in universities, R&D centers and industries in the metal-mechanical sector. The hardness results are also used in the design and development of new materials, products, processes, methods and innovative technologies. Among the existing indentation hardness scales, the most used are Brinell, Vickers and Rockwell ones [1].

The Hardness quantity is also the subject of standardization actions by standardizing bodies which issue international standards (ISO, etc.), regional ones (Mercosul NM, etc.) or national ones – Brazilian standards (ABNT- NBR), American standards (ASTM, etc.) – as well as through the Guide of calibration in hardness, which establishes best practices for obtaining the measurement uncertainty in hardness tests, issued by EURAMET [2], body that consolidates all NMIs (National Metrology Institutes) in Europe.

---

<sup>1</sup> To whom any correspondence should be addressed.



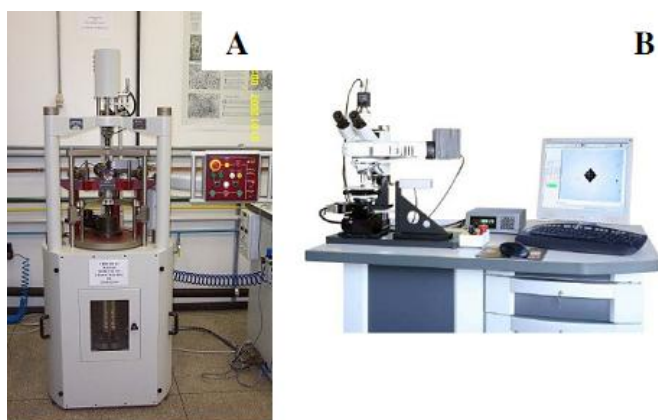
The last paragraph emphasizes the importance of the reliability of the measurement results, reported in metrology as metrological reliability. This feature implies that each measurement shall be related to stated references, or have what is called traceability to national or international standards through an unbroken chain of comparisons each of them having, due to the inherent randomness (variability) of any measurement (since there are several causes for this), some estimates of the involved measurement uncertainties.

To ensure that the metrological reliability is maintained over time, Inmetro's Primary Hardness Standardization Machine has been its main metrological parameters controlled by Shewhart control charts. In case it is necessary any change in the system, no matter the type of event – for example, improvement in terms of accuracy/precision or maintenance of the standardization system –, a system requalification will have to be made.

The objective of this work is to show the procedure for requalification of the primary machine focusing on the application of force during the standardization process of Vickers HV3 hardness scale. The tools used in the requalification process will be described and their importance to keep the standardization system with a suitable metrological quality will be stressed as well. Therefore, if there were no gaps in the traceability chain, the hardness measurements obtained in any durometer (e.g. a hardness testing machine) installed in any industry will provide reliable results. Moreover, they will be considered as traceable to the primary hardness standard of a country (in Brazil, the hardness national standard is Inmetro's Primary Hardness Standardization Machine) and, indirectly, to international standards by international comparisons between Inmetro and other NMIs – under the auspices of the BIPM (International Bureau of Weights and Measures), the Inter-American Metrology System (SIM) or South American Metrology (SURAMET).

## 2. Materials and Methods

The metrological standards used here were the Primary Hardness Standardization Machine (PHSM) – where there is the realization of the quantity hardness by the way of the standardization of Brinell, Rockwell and Vickers hardness scales (figure 1A) – and the Metrological Reference System for Brinell and Vickers Hardness Scales (Gal-Vision), figure 1B. Relating to the latter new image patterns to enable the determination of the Vickers impression diagonal were developed in the image analysis system, so that the hardness measurements and calibrations could be performed with the highest metrological quality.



**Figure 1.** Inmetro's Primary system of Rockwell, Brinell and Vickers hardness scales: (A) Primary Hardness Standardization Machine (PHSM); (B) Metrological Reference System for the Brinell and Vickers Hardness Scales (Gal-Vision).

Objective lenses of magnifications/numerical apertures 2.5x/0.07, 5x/0.12, 10x/0.25, along with a 20x/0.40 lens for adjustment of the Vickers indentation, were used sequentially in the microscope of the Gal-Vision. This procedure was necessary for the measurement of the Vickers diagonals. The Vickers hardness was calculated according to equation 1 [2]:

$$HV = 0.102 \cdot \frac{2F \cdot \sin 68^\circ}{d^2} \quad (1)$$

where:  
 HV – value of Vickers hardness;  
 F – testing force during the standardization, in newtons;  
 d – average length of the diagonals of the indentation, in millimeters.

A 100 N class 00 force transducer was calibrated according to standard ISO 376 [3] was used to check out the applied force during the standardization process. The HV3 hardness values were analyzed before and after the requalification process in such a way it was possible to assure the metrological reliability of both PHSM and Gal-Vision.

International hardness standards ISO 6507-1:2005 [4], 6507-2:2005 [5] and 6507-3:2005 [6] were applied in this study research, which provided the most important parameters of the measurement process that directly influence the Vickers hardness calibration. For comparing before and after requalification's calculations with data coming from hardness measurements, relevant methodologies described in ISO GUM [7] and EURAMET Guide [2] were used. Equations 2 and 3 represent the metrological essence of this requalification, since they are the basis for calculating both the uncertainty regarding the repeatability and the normalized error:

$$u_{rep} = t_{student} \cdot \sqrt{\frac{\sum (\bar{X} - X_i)^2}{n-1}} \cdot \frac{1}{\sqrt{n}} \quad (2)$$

where:  
 $u_{rep}$  – standard uncertainty of measurement due to repeatability;  
 $t_{student}$  – sample size correction factor;  
 n – number of measurements;  
 $X_i$  – any measurement i;  
 $\bar{X}$  – arithmetic mean of measurements.

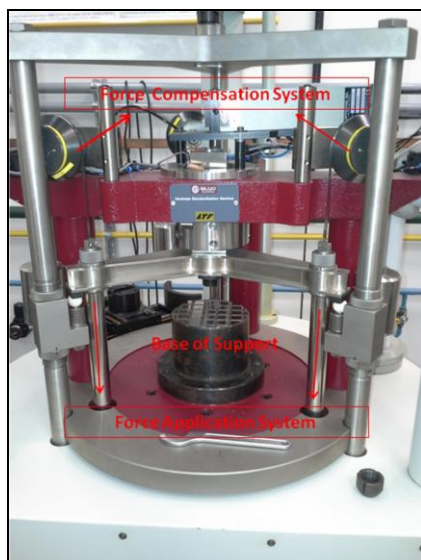
$$E_n = \frac{\bar{X}_i - \bar{X}_{ref}}{\sqrt{U_i^2 + U_{ref}^2}} \quad (3)$$

where:  
 $E_n$  – normalized error;  
 $\bar{X}_i$  – mean hardness HV3 value after the adjustment of the force;  
 $\bar{X}_{ref}$  – mean hardness HV3 value before the adjustment of the force;  
 $U_i$  – expanded measurement uncertainty after the adjustment of the force;  
 $U_{ref}$  – expanded measurement uncertainty before the adjustment of the force.

### 3. Results and Discussion

The results presented below are connected to the PHSM requalification process for the Vickers hardness scale. The scale chosen for the study was the HV3 one, whose scale uses a 3 kgf force. This force is applied to the indenter, which is pressed against the surface of a reference hardness block. This choice was based on the lower force limit of the PHSM, never developed and put in operation

before which means the use of the smallest force (3 kgf) to perform standardization in hardness metrology in Brazil. Thus, the application of force is one of the parameters that need to be monitored because of its high sensitivity to changes in any part of the system. Figure 2 shows a picture of the existing force systems that operate on the PHSM in the standardization process of HV3 hardness scale.



**Figure 2.** Force systems that operate during the standardization of HV3 hardness scale: force compensation and force application ones.

Figure 2 shows that the force application system in case of HV3 is connected to the force compensation system. The application of force of the PHSM is performed by a deadweight system, in other words, gravity acceleration acts directly on the set of stainless steel masses of the system. For HV3 scale, only the framework of the application of forces is acting and its value is equivalent to a 10 kgf selection of masses in the force application system. Thus, to reach a value of 3 kgf it's necessary the use of a force compensation system which, in this case, operates with a load of 7 kgf in the opposite direction to the force application system, resulting in a net force of 3 kgf necessary to perform the HV3 scale.

According to ISO 6507-3:2005, in performing the hardness standardization a machine has to present an absolute percentage error (Error%) less than 0.10% [6]. Thus, adjustments may be necessary in the mass used to generate the force, so that the Error% is in accordance with the limits established by the pertinent standard. Table 1 shows the results for the measurement of PHSM system relative to the application of a 3 kgf nominal force.

**Table 1.** Force verification of the PHSM for a 3 kgf nominal force.

Nominal Force/N	Force/N	Force/kgf	Error/kgf	Error%/%
29.362	29.25726	2.989252	-10.7482	-0.358
29.362	29.25776	2.989303	-10.6972	-0.357
29.362	29.25876	2.989405	-10.5951	-0.353
29.362	29.25976	2.989507	-10.4931	-0.350
29.362	29.25926	2.989456	-10.5441	-0.351

It can be observed in table 1 that the Error% values were out of the  $\pm 0.10\%$  limits allowed by the ISO 6507-3:2005 [6]. In fact, there is a case of negative Error% i.e. it means there is a lack of masses to attain the nominal force value. So, it was concluded that the best way to increase the metrological quality of the force application process for the standardization of HV3 scale was the addition of specific amounts of tiny masses to the force application system of PHSM. A detailed study, comprising five different conditions C1 to C5, was conducted by the addition of mass increments to the PHSM as shown in Table 2.

**Table 2.** Study of the force applied in the PHSM for a 3 kgf nominal force by using tiny masses adjustment.

Condition	Nominal Force/kgf	Adjustment/g	Force/kgf	Error%/%
C1	3	0	2.9894	-0.354
C2	3	9	2.9985	-0.049
C3	3	10	2.9996	-0.015
C4	3	11	3.0006	0.019
C5	3	15	3.0045	0.150

In table 2, the reported values of nominal forces and Error% were obtained from an average of five measurements. Accordingly, it can be noted that condition C3 presented the minor Error% value, being considered the most appropriate for the requalification process of the HV3 hardness scale at PHSM. Figure 3 shows graphically the effect of the addition of masses to the PHSM on the Error% related to the force applied during the standardization of HV3 scale.



**Figure 3.** Error% relative to the measurements performed in the conditions C1, C2, C3, C4 and C5.

Figure 3 is a corresponding graph of Table 2. It can be noticed that the mass increments promotes a trend that is represented by the vertical red arrow, in the direction to the decrease of the absolute value of the Error%. So, through the analysis of figure 3, it can be observed that the condition C3 is the one that has the lowest absolute value for the Error%. Thus, during the standardization made to HV3 scale at PHSM it was necessary the addition of a 10 g mass to the force application system.

On finishing the above analysis to determine the best condition for the application of force in the PHSM, the first part of the task of requalifying study was realized. The next and last step was a verification of the performance of PHSM related to standardization process itself under this new

operating condition. It means reference hardness blocks calibrated before the requalification were recalibrated and their results compared to low, medium and high hardness ranges of HV3 scale.

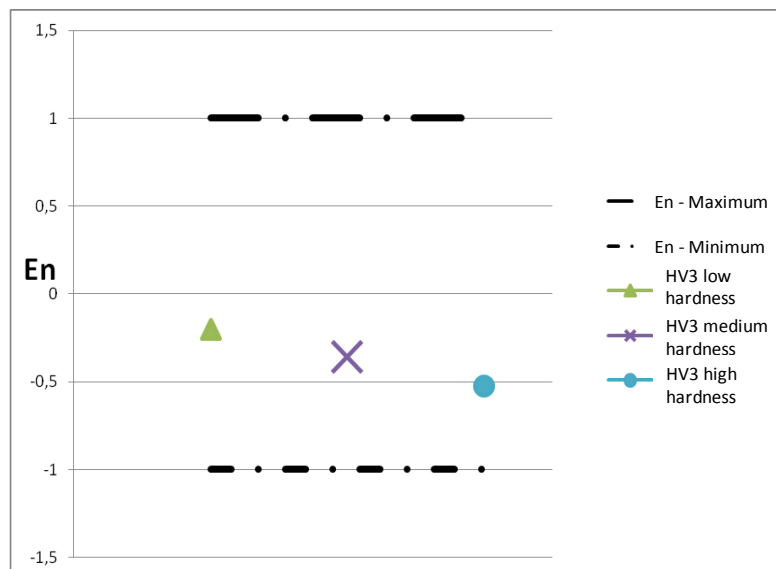
Table 3 presents the average values of hardness, standard uncertainty of measurement due to repeatability and expanded measurement uncertainty to the low, medium and high hardness ranges of HV3 scale before and after the requalification of PHSM.

**Table 3.** Performance study of PHSM during the standardization of HV3 scale.

Parameters	HV3 low hardness		HV3 medium hardness		HV3 high hardness	
	Before	After	Before	After	Before	After
Average hardness	131.7	131.0	568.2	565.7	848.2	844.9
Standard uncertainty of measurement due to repeatability	0.8	0.8	3.1	1.2	2.5	1.4
Expanded measurement uncertainty	2.5	2.6	6.4	3.0	5.3	3.4

In table 3 it was found that for low HV3 hardness, the force requalification process did not cause any significant changes in hardness since the hardness indentation size is big, so its diagonals were easily read. As a consequence, the related expanded measurement uncertainties, before and after the HV3 requalification, had nearly no changes. However, for medium and high ranges of HV3 hardness - that have smaller indentation sizes - it can be said the requalification process was effective and improved the metrological quality of the measurements, since it was possible to obtain a reduction in the expanded uncertainty. Evidence of this comes from the decrease of 53% (from 6.4 to 3.0 HV3) for the medium HV3 hardness range and 36% (from 5.3 to 3.4 HV3) for the high HV3 one.

Figure 4 shows the review of the normalized errors with respect to standardization of HV3 scale for low, medium and high hardness ranges before and after the force adjustment process.



**Figure 4.** Normalized errors for low, medium and high HV3 hardness ranges.

Figure 4 depicts all calculated normalized errors taking into account the obtained results before and after the force requalification process: they are within the normalized error limits of 1 and -1. In other words, this result showed that not only the requalification promoted an improvement in the metrological quality of PHSM but also its standardizing measurements are still reliable. In metrological terms, these results indicate that both the PHSM and the Gal-Vision metrological standards have the necessary prerequisites for the HV3 scale standardization, since there was a high similarity between the normalized errors before and after the force adjustment for lower, medium and high hardness ranges. Then, the methodology applied in this work can eventually be applicable to others hardness scales in their requalification processes.

#### 4. Conclusions

- (i) This work showed the metrological methodology applied in the requalification of the Inmetro's Primary Hardness Standardization Machine for low, medium and high hardness ranges of HV3 scale.
- (ii) Results in terms of normalized errors obtained with the requalification of the PHSM for HV3 Vickers hardness scale were satisfactory, since they were within the limits of 1 and -1.
- (iii) Inmetro's PHSM is suitable for performing the standardization of HV3 scale in Brazil, since the % relative error to the nominal applied force was less than 0.10%.
- (iv) On comparing the expanded uncertainty of the HV3 scale before and after requalification it yields an increase in the metrological quality of the results for middle and high HV3 hardness ranges because there was a reduction in the uncertainty of results for both hardness ranges.
- (v) The normalized error, as a result of a comparison of measurements reliability before and after the requalification process, has shown a high similarity between them. This was an evidence that the parameters involved in the standardization process remain under control at the end of the requalification process.

#### 5. Acknowledgment

The authors thank to FAPERJ (Research and Development Support Foundation of Rio de Janeiro State), for the research support grant for the technologist Celso Ricardo da Silva Azeredo, and also to Inmetro (National Institute of Metrology, Standardization and Industrial Quality) where all of the researchers of this study work in the Force Laboratory (which deals with areas of force, torque, hardness and impact), for supporting the development of this work.

#### 6. References

- [1] William D. Callister, Jr. "Materials Science and Engineering – An Introduction". New York/USA: John Wiley & Sons, Inc., 7<sup>th</sup> Edition, 2007.
- [2] "Calibration Guide EURAMET/cg-16/v.01: Guidelines on the Estimation of Uncertainty in Hardness Measurements". Braunschweig/Germany: European Association of National Metrology Institutes, 2007.
- [3] ISO 376:2011 "Metallic materials – Calibration of Force-proving Instruments Used for the Verification of Uniaxial Testing Machines", Geneva/Switzerland: International Organization for Standardization, 2011.

[4] ISO 6507-1:2005 “Metallic materials – Vickers hardness test – Part 1: Test method”. Geneva/Switzerland: International Organization for Standardization, 2005.

[5] ISO 6507-2:2005 “Metallic materials – Vickers hardness test – Part 2: Verification and Calibration of Testing Machines”. Geneva/Switzerland: International Organization for Standardization, 2005.

[6] ISO 6507-3:2005 “Metallic materials – Vickers hardness test – Part 3: Calibration of Reference Blocks”. Geneva/Switzerland: International Organization for Standardization, 2005.

[7] JCGM Member Organizations (BIPM, IEC, IFCC, ILAC, ISO, IUPAC, IUPAP and OIML). “JCGM 100:2008 – GUM 1995 with minor corrections – Evaluation of Measurement Data – Guide to the Expression of Uncertainty in Measurement”. Sèvres/France: Joint Committee for Guides in Metrology/Bureau International des Poids et Mesures, 2008.