Assuring the quality of results of test hardness IRHD: IPT’s case study

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Abstract. This paper presents the experience of the Laboratory Trees, Woods and Furniture - LAMM in calibrating the durometer IRHD and assuring the quality of its test results, since there are no Certified Reference Material and laboratory in the Brazilian Calibration Network that can calibrate the equipment. To solve this problem, the IRHD hardness (N method) was quantified in three ways: a) by measuring the modulus of elasticity of the material, b) by measuring the depth the sphere entered the material and c) the durometer’s direct reading. With the IRHD hardness measured by accepted international standards techniques, it was possible to evaluate the accuracy of the test results that assured the calibration of the equipment.

1. Introduction

The devices and materials involved in the characterization tests and performance evaluation of melamine laminated flooring and furniture, require the use of polymeric materials with specified hardness values. Therefore, it is necessary to know these values in order to check for their compliance with the requirements of the related standards.

To perform this test the Laboratory Trees, Wood and Furniture - LAMM from the Technological Research Institute of São Paulo State S.A - IPT, acquired an IRHD hardness tester in the range 30 IRHD to 100 IRHD. After purchasing the equipment, the LAMM faced the problem of ensuring the metrological reliability, since there is no laboratory in the Brazilian Calibration Network (RBC) that could calibrate the equipment.

In addition, there isn’t a producer of certified reference materials in Brazil besides, the purchase abroad does not guarantee prompt delivery. As a result, to solve the calibration problem of the equipment and to ensure the quality of the test results, the LAMM prepared samples of a selected polymeric material and subjected them to tests from the following accepted international standards: ISO 48: 2010 [1], ISO 18898: 2012 [2] and ISO 7743: 2011 [3], applicable to different polymeric materials. All tests were done under controlled conditions of temperature and relative humidity, according to the criteria established at ABNT NBR 13966: 2008 standards [4] and ABNT NBR 14833-1: 2009 [5].

With the data collected from the determination of the modulus of elasticity of the material, the measurement of the depth of indentation of the sphere and the direct reading on the tester, the results
were compared via a statistical analysis [6, 7] in order to check for the calibration of the equipment and to verify the quality assurance of the results [8].

2. Objective
This article aims to show the experience of the LAMM in the calibration procedure of an IRHD tester and to demonstrate the quality assurance of the results of an IRHD hardness test.

3. Methods
From a piece of polymeric material six circular shaped samples were cut with a diameter of 29.0 mm and thickness of 12.5 mm, they were conditioned at a relative air humidity of (50 ± 5) % and a temperature of (23 ± 2)°C for a period of 7 days.

3.1 Definition of hardness IRHD
According to ISO 48: 2010, hardness IRHD by method N is a quantity defined as a function of the depth of indentation, expressed as (1/100) mm, of a steel sphere with a diameter of (2.50 ± 0.01) mm, on a polymeric material initially subjected to a force of (0.30 ± 0.02) N, under the action of an additional force of (5.40 ± 0.01) N. The relationship between the depth of indentation of the sphere ‘D’ in (1/100) mm and its hardness, is the IRHD shown in table 3 - Conversion of D values of IRHD for the N method, from the standard ISO 48 [1].

3.2 Weight and diameter of the sphere tester
The diameter of the sphere was measured and the weight of the device was determined in order to verify whether they met the requirements of the standard ISO 48: 2010 [1].

3.3 Calibration via modulus of elasticity
In this IRHD hardness calibration method it was used Equation 1 from ISO 48: 2010 [1], which calculates the indentation depth of the sphere and allows to express the value of IRHD hardness with the help of table 3 - Conversion of D values of IRHD for method N of standard ISO 48 [1], forementioned.

\[
D = 61.5 \times 10^{-0.84} \left( \frac{F_{in}}{E} \right)^{0.74} - \left( \frac{F_c}{E} \right)^{0.74}
\]  

(1)

where:

- \( D \) = the differential indentation (1/100) mm;
- \( R \) = the radius of the ball (mm);
- \( F_{in} \) = the total indenting force (N);
- \( F_c \) = the contact force (N);
- \( E \) = specimen’s modulus of elasticity (MPa).

As previously mentioned, the IRHD hardness is attributed to the indentation of a sphere of 2.50 mm in diameter, under an contact force of 0.30 N in addition to a force of 5.40 N, resulting in the total indenting force of 5.70 N.

The determination of the modulus of elasticity (E) was in accordance with ISO 7743: 2011 [3], with a deformation up to 5%.

Having obtained the value of E, equation (1) was used to estimate D namely, the relative indentation. Referring to the value of the D from Table 3 - Conversion of D values of IRHD for method N from standard ISO 48, the corresponding value of IRHD hardness was obtained.

3.4 Calibration via the depth of indentation of the sphere
This test was based on the criteria established in ISO 48: 2010 [1] and ISO 18898: 2012 [2].

The device used for measuring the depth of indentation of the sphere consisted of a photographic camera with a calibrated scale and with standard of reference, attached to a digital microscope Dino-Lite Pro, model AM413T PRO. The depth of indentation was determined by taking two photos with the still camera and measuring reference points before and after the application of the force.

With the values of the depth of indentation of the sphere, the IRHD hardness was obtained, according to the standard ISO 18898: 2012 [2].

3.5 Determination of hardness IRHD N with tester
The determination IRHD hardness was performed directly, according to the criteria of ISO 48: 2010 [1], using the IRHD hardness tester N.

3.6 Precision Experiment
To determine the precision under the condition of repeatability, according to ISO 5725-4: 1994 [6], IRHD hardness tests were carried out on eight samples with three replicates, using the hardness tester IRHD N.

4. Results

4.1. Weight and diameter of the sphere tester
The statistics of the measurements on ball diameter and weight of the device, are presented in table 1.

| Statistics a | Diameter (mm) | Weight (N) |
|-------------|---------------|------------|
| Average     | 2.497         | 5.396      |
| Standard deviation | 0.00063       | 0.00005    |
| Reference value (ISO 48) | 2.50         | 5.40       |
| Error = average - reference | - 0.003      | - 0.004    |
| Tolerance (ISO 48) | ± 0.01       | ± 0.01     |

aResults of 10 measurements.

4.2. Hardness IRHD N
The IRHD N hardness results calculated from the modulus of elasticity and the depth of indentation of the sphere and those obtained directly from the durometer readings according to the methods presented, respectively, in items 3.3, 3.4 and 3.5 are presented in table 2.
Table 2. Results of IRHD hardness calculated from the modulus of elasticity, the depth of indentation of the sphere and those obtained directly from a tester.

| Specimens | Modulus of elasticity ($E^a$) | Measurement of the depth of indentation of the sphere ($D^b$) | Hardness IRHD N | Hardness IRHD N | Hardness IRHD N |
|-----------|-------------------------------|-------------------------------------------------------------|-----------------|-----------------|-----------------|
|           |                               |                                                             | $D^c$           |                  |                  |
| 1         | 4.64                          | 57                                                          | 67.1            | 68              | 61.5            |
| 2         | 4.34                          | 60                                                          | 65.5            | 68              | 61.5            |
| 3         | 4.54                          | 58                                                          | 66.6            | 72              | 59.7            |
| 4         | 4.57                          | 58                                                          | 66.6            | 72              | 59.7            |
| 5         | 7.10                          | 42                                                          | 75.8            | 48              | 72.2            |
| 6         | 4.77                          | 56                                                          | 67.6            | 68              | 61.5            |
|           | Average                       |                                                             | 68.2            | 66              | 62.7            |
|           | Standard deviation            |                                                             | 3.0             | 9.0             | 4.7             |
|           |                               |                                                             | 3.8             |                  |                  |

Legend:

$^aE =$ modulus of elasticity of the specimen (MPa).

$^bD =$ differential indentation calculated from in the equation using the modulus of elasticity (1/100) mm.

$^cD =$ differential indentation obtained from direct measurement (1/100) mm.

The results of the statistical analysis of the IRHD hardness obtained via the methods of the modulus of elasticity and the measurement of the depth of indentation of the sphere when compared with those obtained directly from the tester, are shown in table 3.

Table 3. Results of the IRHD hardness obtained via the methods of the modulus of elasticity and the measurement of the depth of indentation of the sphere.

| Statistics $^a$ | Module of elasticity | Direct measurement of the depth |
|-----------------|----------------------|---------------------------------|
| Mean difference | 4.8                  | -0.8                            |
| Standard deviation | 0.864              | 1.343                           |
| $t$ calculated  | 13.462               | -1.399                          |
| Degrees of freedom | 5                  | 5                               |

| Results $^a$ | Significant difference | Non significant difference |
|--------------|-------------------------|----------------------------|
| $t$ critical | 2.015                   |                            |

$^a$ Difference = readings from the method - readings from the tester

Figure 1 shows the comparison among the hardness results estimated by the proposed methods and their respective regression equations.
4.3. Experiment of the Precision of the durometer IRHD N

The results of the test for determining hardness IRHD obtained with the IRHD hardness tester, to evaluate the repeatability are presented in Table 4.

| Specimens | IRHD | Variance | Sr² | Repeatability |
|-----------|------|----------|-----|---------------|
| 1         | 61.4 | 61.4     | 61.0| 61.3          | 0.05         |
| 2         | 60.5 | 60.7     | 60.6| 60.6          | 0.01         |
| 3         | 61.2 | 61.4     | 61.3| 61.3          | 0.01         |
| 4         | 60.7 | 61.1     | 60.8| 60.9          | 0.04         |
| 5         | 61.0 | 61.3     | 60.6| 61.0          | 0.12         |
| 6         | 61.3 | 61.3     | 61.7| 61.4          | 0.05         |
| 7         | 60.7 | 61.0     | 61.3| 61.0          | 0.09         |
| 8         | 61.1 | 61.4     | 61.1| 61.2          | 0.03         |

\[
\text{Sr}^2_a = 0.05 \\
\text{Repeatability} = 0.22
\]

\[
\text{Compliance to Equation 9, standard ISO 5725-4 [6].}
\]

\[
\text{Compliance 8, standard ISO 5725-4 [6].}
\]

The repeatability obtained for the durometer of IRHD was of 0.22 hardness IRHD unit.
5. Discussion
It has been demonstrated the suitability of the tester by verifying the compliance of the results obtained by the weight (5.40 N) and the sphere diameter (2.50 mm), shown in table 1, to the limits specified in ISO 48: 2010 [1].

The manufacturer of the tester sent, at the time of acquisition of the equipment, a calibration certificate nº 10620, of 25/08/2011, issued by the Deutscher Kalibrierdienst DKD, calibration label: DKD-K-16501.

In this certificate the uncertainty in the detected sag was of 0.3 hundredths of a millimeter, which corresponds to about 0.15 to 0.18 unit the IRHD hardness, based on table 3 of the ISO 48 standard for IRHD hardness, in the range of 53-66.

In table 2 it can be seen a non-significant difference between the values determined of the hardness IRHD with a durometer (63.4) and the calibration by measuring the depth of indentation of the sphere (62.7).

This non-significant difference can be explained by the fact that between the two measurements, according to the method of determination, the only variable that was changed was the way of measuring the indentation of the sphere.

In the case of calibration via modulus of elasticity of the material, see table 2, the difference was significant, because this method tends to estimate higher hardness values them the ones obtained with the tester.

On the other hand estimation of the modulus of elasticity (E) of the polymeric material is influenced by many variables that contribute to the uncertainty of the result such as: force, deformation, specimen dimensions, contact surface lubrication and positioning of the specimen.

Regarding to the results of the precision experiment, the repeatability of 0.22 in the IRHD hardness obtained with the tester, is compatible with the value expressed in the Calibration Certificate of the equipment.

6. Conclusion
From the results obtained it can be concluded that:

a. The determined values of the sphere diameter and the load of the device IRHD hardness tester are in accordance with ISO 48 [1], Method N.

b. The estimated values of IRHD hardness obtained via the modulus of elasticity method, showed significant difference from those obtained with IRHD hardness tester.

c. The results of hardness obtained with the IRHD hardness tester were not significant from those estimated via the measurement of the depth of indentation of the sphere method.

d. The method of measuring the depth of indentation [1] to calibrate IRHD tester results, showed to be adequate, since it allowed quantifying the displacements at different loading points by optical means without physical contact.

e. It was possible to demonstrate the quality assurance of the results of the IRHD hardness test.

f. New test procedures can be developed and offered internationally to the technological environment in order to improve the services to the users of standardized materials.

7. References
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[3] International Standard Organization 2011 ISO7743 – Rubber, vulcanized or thermoplastic – Determination of compression stress-strain properties (Geneva, ISO) p 22
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