ANALYSIS ON THE PERFORMANCE OF MECHANICAL TEST LABORATORIES BASED ON A PROFICIENCY TESTING

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Abstract. *This work presents a study on technical performance of mechanical testing laboratory, located in Rio de Janeiro State, considered as important generators of information for the characterization process of metallic materials properties applied in industry, be them accredited, or no, included to INMETRO - National Institute of Metrology, Normalization Industrial and Quality. Inside of available group tests for this end, opted to focus those that, due to nature and application of materials, they are, more frequently, used. Therefore, the results, object of evaluation, they are those obtained in tests for the determination of mechanical property hardness (Rockwell, Brinell and Vickers). As reference for analysis, the established requirements were adopted in the norm ABNT ISO/IEC 17025:2001, in the which they are outstanding those that have more impact, on laboratories results. As instrument for the obtaining of data was drifted, and developed a Proficiency Testing (PT), base on ABNT ISO GUIDE 43-1:1999, associate to the Youden Method that it will supply the statistical elements for analysis. This treatment method of data processing, uses a 95% confidence ellipse, that it is for identification of laboratories whose results, in principle, will evidence, or no, a special performance situation, besides considered its positioning in the specific quadrants. In the development of the PT, actions will be accomplished in the sense of characterization of reference materials, invitation and selection of the participant laboratories, perform of measurements, statistical treatment of data analysis. It will be possible to detach the laboratories, in function of your metrology performance, being evidenced, besides, the possible causes. The conclusions of this study will be marked strongly by the analysis of the variability results of laboratories and its influence on the reliability of the information generated in the relative tests to the mechanical properties of metallic materials.*

Keywords*: proficiency test, hardness test, technical performance, mechanical test laboratories, Youden Methodology*

1. Introduction

It comes from long date the importance of materials science, real revolutions happen when if discovery new materials. Everything this, leans on in experiments that make possible the knowledge more and more detailed of the materials properties, and it serves as an immense technological database with contemplation in several areas of the pure science or technological applications in industry.

Among the several properties of the materials, the mechanics are highlighted (Fig. 1), that it comprehends aspects as resistance, elasticity, plasticity, fatigues, fractures and deformation. Resistance (tensile strength, compression, torsion, flexion, fatigue, bend, impact and shear strength), elastic limit, ductility, resilience, tenacity and hardness are some of the mechanical properties whose applications are made necessary in the areas more common of the engineering as: civil, mechanics, naval, metalworker, automotive and warlike; and also in the more advanced as aerospace, biotechnology and nanotechnology.

The properties mechanics are all of the measurable characteristics, in other words, quantitatively determined, of the materials or finished products, when these are submitted to mechanical loads. Under the application of these loads, the materials react being deformed, elastic or plastically, and in some cases fracturing in agreement with the influence of the temperature, the environment the one that is exposed, the time and the speed of load application.

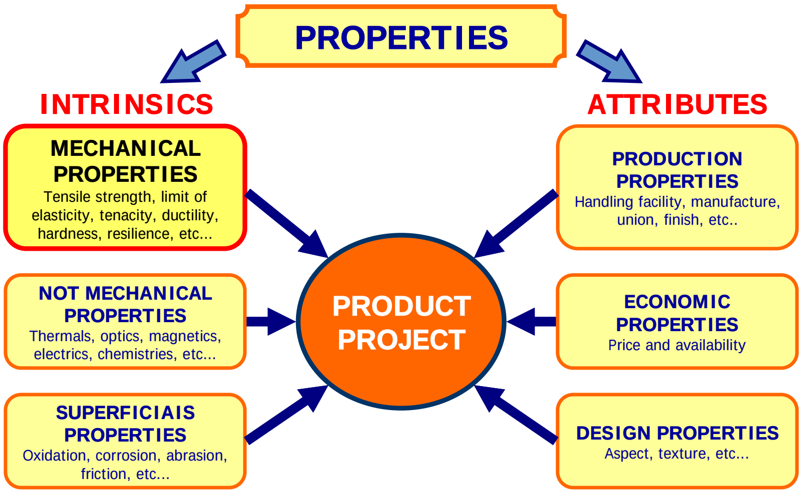


Figure 1. Importance of materials properties to development of a product, especially the mechanics.

For the determination of the properties mechanics several tests are performed, that it demands an appropriate infrastructure and resources, such as: machines, instruments, reference materials, human resources (qualified professionals) and methodology (norms, procedures, good practices), localized its called of *Mechanical Tests Laboratories* - MTLs. It main functions are: to measure, to examine, to test, to determine characteristics and performing of materials, structures or products, when submitted to mechanical loads (ABNT ISO/IEC 17025, 2001). There are diverse models of MTLs, in this study it was divided in four groups: industries laboratories (INLs), Services Provider (SPLs), Researches Centers (RCLs) and Teaching Institutions (TILs).

For safety and economical reasons, in engineering projects, tests are used in one or more parts or a determined component is submitted to load mechanical, instead of the whole structure. Keep big similarity with those that it is applied in a real situation of service. For this, samples of these materials are used with normalized dimensions. These samples to they are rehearsed by MTLs, they generate results, and these values are compared to specified values to material. The data, obtained at laboratory, don't guarantee the performance in service; however, they are important indications of material conformity with a group of mechanical properties, that the experience has been showing, continually, satisfactory performance in service.

Out of the tests that can be executed by MTLs (Fig. 2), it emphasizes for a larger industrial application, the following tests: tensile strength, Charpy impact test, Brinell, Rockwell and Vickers test.

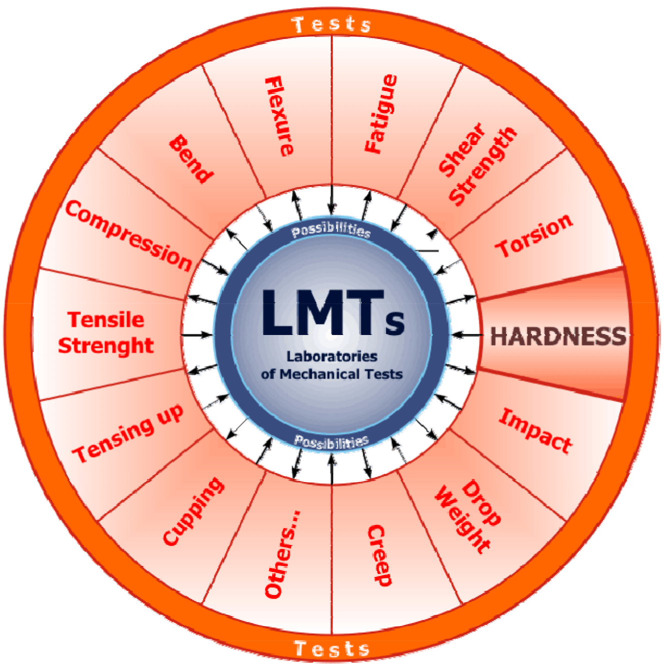


Figure 2. Types more common of mechanical tests performed by MTLs, highlight for hardness tests.

2. The Chosen Test and the Proficiency Testing

As shown previously, MTLs perform many types of tests (Fig. 2). For a wide analysis of performances these laboratories, would be necessary the analysis of all those tests. But, for obvious restrictions to this research type, such as: time, cost, distance and human resources, it was necessary to reduce the scope of this research for just one test. And choice for development of the PT is due, as seen in the previous item, and it will be seen to precede, the importance of mechanical property hardness and the largest industrial application of the tests of hardness. Besides needing of equipment (durometer, Fig.3), relatively cheaper, if compared to the necessary ones for the other tests (Fig. 2), counting so, with a bigger number of available laboratories.

They were already pre-registered, approximately seventy laboratories, out of INLs, SPLs, RCLs and TILs, able to participate in the program, only in the State of Rio de Janeiro / Brazil, that will be the actuation area of this study.

Above works (Oliveira, 1990), (Leete and Friday, 1998) and norms (ABNT ISO/IEC Guide 43-1 and 2, 1999), (ABNT NBR ISO/IEC 17025, 2001), they already indicated the course to follow for performance analysis of MTLs and increase of the metrological reliability. And recently, in the year 2003, Brazilian Government introduced the PITCE - Politics Industrial, Technological and External Trade, that through the Public Call MCT/FINEP - Traverse Action - TIB-06/2005, Line 3; ratifying the importance of this program type for industrial development of country. It will be applied until the end of 2005, R$ 2,680,000.00 (two million, six hundred and eighty thousand Real)

2.1. Hardness Tests

The hardness test is used in wide scales in industry, so much in specification, as in control of materials characteristics. It is also in research and development area (R&D), as example, to verify the modification of material properties after a thermal treatment.

Even tough usually to be considered as resistance to the plastic deformation, the material hardness is not interpreted in the same way by different professionals (Souza, 1982). In some cases, also it is considered as resistance to risk, to penetration, to abrasion and deformation. Through the several durometer types (Fig. 3), different tests types are performed (Fig. 4), in agreement with the concepts mentioned above.

This test presents various advantages: fast execution and relative facility, if compared with other tests (Fig. 2); parts or pieces, finished or no, and any size or forms, they can be tested, although to be considered a destructive test (Souza, 1982), a small indentation (sphere Ø2,5mm, or diamond conical indenter), sometimes, it doesn't discard the use of tested part; practical inspection device and control of quality, in the reception and materials expedition; and a lower cost.

The Brinell, Rockwell and Vickers tests are the main methods and with bigger application in actuation area (Rio de Janeiro / Brazil).

Font: Instron Catalog (2004), page 6.

Figure 3. Instron Durometer Series 2000.

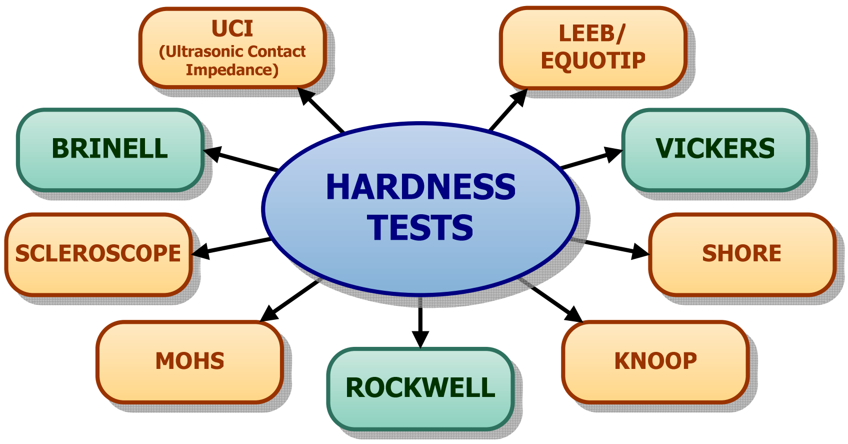


Figure 4. Types of hardness tests highlight for methods focused in this paper.

2.1.1. Brinell Test

Dr. J. A. Brinell invented the Brinell test in Sweden in 1900. This method consists of pressing a sphere of steel or carbide, denominated indenter, into the sample. Staying the load applicator during a short interval of time, it is produced of this mode an indentation in format of a spherical cap.

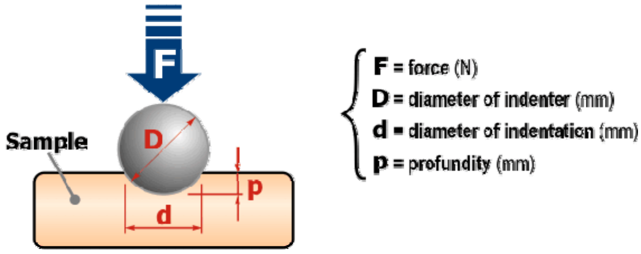


Figure 5. Scheme of Brinell test.

The Brinell hardness number is a function of the test force divided by the curved surface area of the indent, Eq. (1). The indentation is considered to be spherical with a radius equal to half the diameter of the ball. The average of the two diagonals is used in the following formula to calculate the hardness, and the number *0,102* it is due to use of SI units.

$$HB = \frac{2 \cdot F \cdot (0,102)}{D \cdot \left(D - \sqrt{D^2 - d^2}\right)} \quad (1)$$

Combinations of loads, between 29,420 N (3,000 kgf) and 49 N (5 kgf), and indenters (Ø10; Ø5; Ø2.5 and Ø1mm), they allow tests in a large number of materials.

The Brazilian norm ABNT NBR NM 187-1 (1999) specifies the measurement method of hardness Brinell for metallic materials, but other including norms exist, as: ASTM E10-01e1 (2001) and ISO 6506 (1999). Therefore, the specification of the normative criteria is basic requirement for obtain of useful values to industrial job.

Besides the test method, the parts 2 and 3 of the norm ABNT NBR NM 187 (1999) defines relative parameters to the characteristics of the optic instruments, indenter and durometer, it also supplies criteria for durometer and reference materials calibration, in agreement with the concepts of accuracy and precision.

Note: In the US, previous standards allowed a steel ball and had an S designation. Steel balls are no longer allowed.

2.1.2. Vickers Test

This method was developed by Smith and Sandland in England is 1925, based on test of Brinell hardness. And it looked for to solve the limitation of this test: deformation of the sphere when tested materials of high hardness, with the use of a single diamond indenter, in format of square base pyramid with angle of 136° between the opposite faces (Fig. 6). It uses loads between 0.098 N (1 gf) and 980 N (100 kgf), getting to measure hardness of the materials microstructure.

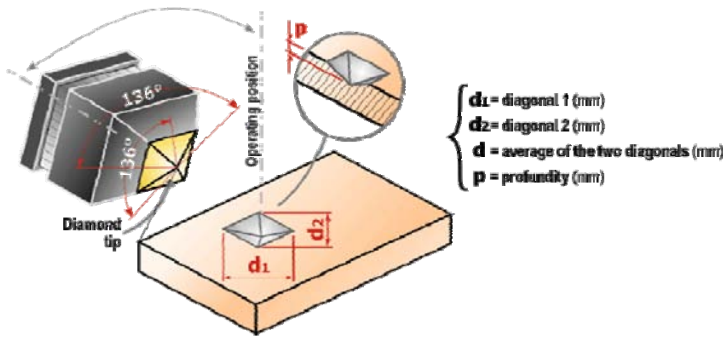


Figure 6. Scheme of Vickers test, detail for diamond pyramid indenter.

In this test, the Vickers hardness number also is a function of the test force divided by the total area of the indent, Eq. (2).

$$HV = 0,189 \cdot \frac{F}{d^2} \quad (2)$$

The Brazilian norm ABNT NBR NM 187-1 (1999) specifies the measurement method of Vickers hardness for metallic materials, and the parts 2 and 3, criteria for durometer and reference materials calibration. They also exist: ASTM E92-82e2 (2003), ASTM E384-99e1 (1999) and ISO 6507 (1997).

2.1.1. Rockwell Test

Presented for Stanley P. Rockwell in 1922, this test consists of penetrating the sample, with an appropriate indenter and a short interval of time. The resulting Rockwell number represents the difference in depth from the zero reference position as a result of the application of the major load, just considering the produced plastic deformation (Fig. 7). Compared to the other two methods, it is sufficiently simple and fast in its execution, because it eliminates the measurement stage (diameters or diagonals) of indentation. The result is read directly in the durometer dial.

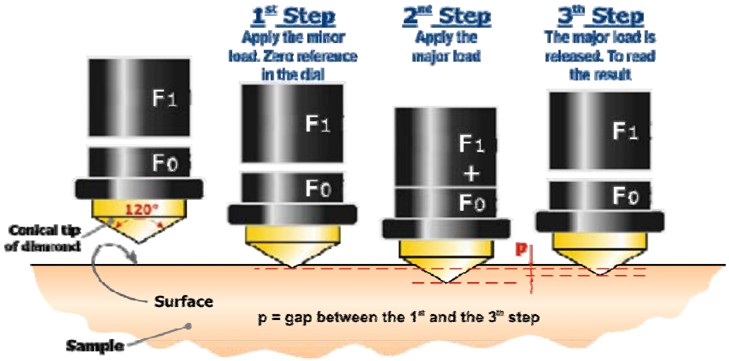


Figure 7. Steps for performing Rockwell test.

It is divided in two types: Normal Rockwell, with minor load of 98 N (10 kgf) and major loads of 588; 980 and 1,470 N (60; 100 and 150 kgf, respectively) and Superficial Rockwell, with minor load of 29.4 N (3 kgf) and major loads of 147; 294 and 441 N (15; 30 and 45 kgf). For each type, presents a total of fifteen scales, combining their three possible loads with their five indenters: diamond cone with angle of 120°; steel spheres with diameters of 1.59 mm (1/16"), 3.18 mm (3/8"), 6.35 mm (1/4") and 2.7 mm (1/2"). The used scales of normal Rockwell test are: A, B and C.

The Brazilian norm ABNT NBR NM 146-1 (1998) specifies the measurement method of Rockwell hardness for metallic materials, and the parts 2 and 3, criteria for durometer and reference materials calibration. They also exist: ASTM E18-05e1 (2005) and ISO 6508 (1997).

Note: Nowadays, there are Brinell and Vickers durometers capable to eliminate the step of manual measurement, through digital image acquisition system and electronic measurement, showing the result directly in your LCD viewer or computer screen.

2.2. Proficiency Testing (PT)

The PT is an activity in that it participate various laboratories that through the made measurements and appropriate statistical data treatment, it will make possible the obtaining an information series about technical performance on each participant (Oliveira, 1990).

In the specific case of this work, the PT will just be a tool for general analysis of MTLs performance, and their subsequent conclusions will have a restricted validity to the time of it performed. This study type if it doesn't perpetuate along the years, because, only in the last ten years, already all were reformulated from the pertinent and applied norms to MTLs, and also, it is more and more fast the evolution of equipments and applicable instruments. Therefore, this study should serve as base to know the current state of the art, for maybe, to propitiate new reformulations and evolutions.

2.2.1. Development and Characteristics

The present PT will be applied MTLs qualified to perform hardness tests in metallic materials and it will follow recommendations of norm ABNT ISO/IEC Guides 43-1 (1999).

It presents the following objectives:

1. Evaluation of laboratories performs participants in what refers the hardness measurement in metallic materials, in other words, to evaluate the MTL ability in performing the proposed tests in a competent way;
2. To allow a self-knowledge on the part of the participants, being possible to identify no-conformities and to begin corrective actions;
3. To do a correlation between the competences technique and managerial according to the requirements of the norm ABNT NBR ISO/IEC 17025 (2001).

MTLs that they answer affirmatively to the invitation, that should happen in beginning of June, 2005, will receive a visit previously schedule, for interview with the responsible for MTL. In this opportunity, also two samples will be taken to be tested and larger information as for the test procedure and pertinent norms. Figure 8 illustrates the flowchart of the PT.

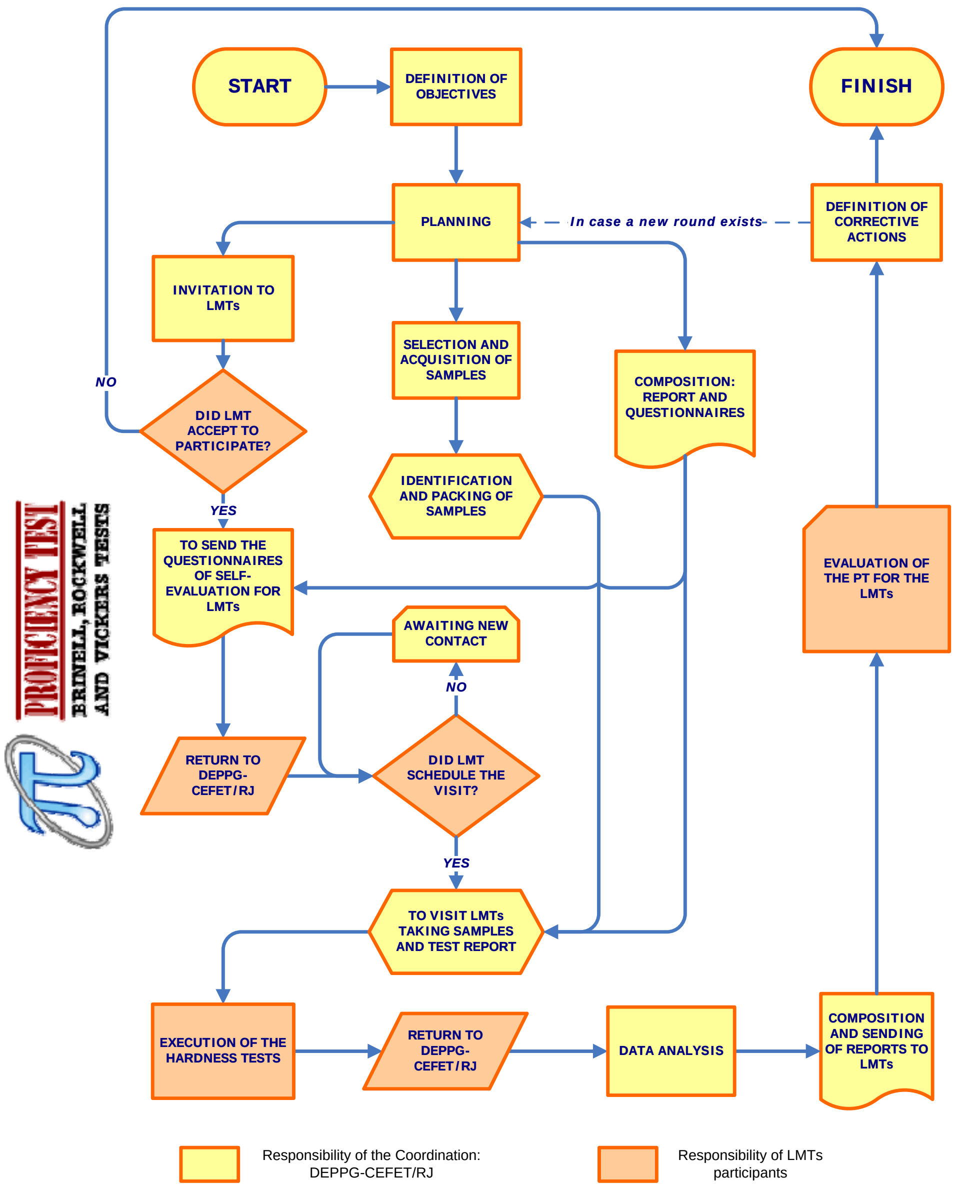


Figure 8. Flowchart of the Proficiency Testing - PT

Each participant will schedule and it will execute in agreement with the procedure, the requested tests.

It will be made the processing and data analysis in agreement with the Youden Methodology (ABNT ISO/IEC Guides 43-1, 1999). This is a graphic method elaborated in function of 95% confidence ellipse.

Each MTL will be identified by a code, in way to maintain to total confidentiality about the participants' identity. And it will be, later, sent by DEPPG/CEFET-RJ a report in which each participant MTL, identified for your code, it will visualize your performance in relation to other participants.

2.2.1. Proficiency Testing Results

In agreement with the established schedule, it is hoped for July of 2005, even to count with the return of the tests results performed by MTLs, and until September of 2005, to be sending the PT reports to participants.

For the choice of statistical tools and validation of computational package, it was collected data (Tab. 1) of PT performed by Oliveira (1990), that it resulted in the choice of the program Statistic under license of StatSoft Inc., capable to operate satisfactorily the whole processing of data, statistical calculations and generation of 95% confidence ellipses (Figs. 9 and 10).

Table 1. The averages had obtained in the Brinell tests, with sphere Ø10 mm and load of 2,942 N (3,000 Kgf) in the PT.

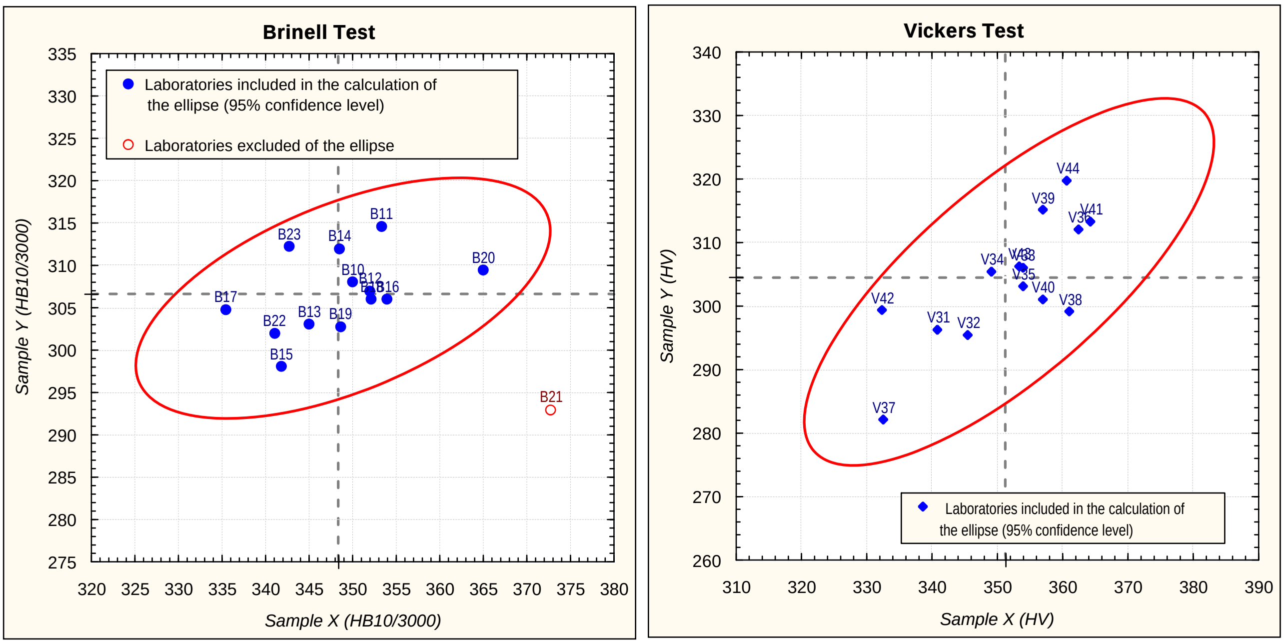
MTL Code	→	B10	B11	B12	B13	B14	B15	B16	B17	B18	B19	B20	B21[(1)]	B22	B23
Sample number	→	X26	X19	X11	X01	X10	X20	X05	X30	X15	X10	X06	X18	X24	X16
Average (HB10/3000)	→	350	353,3	352,0	345	348,5	341,8	354	335,4	352,2	348,6	365	372,8	341,0	342,7
Sample number	→	Y02	Y03	Y22	Y17	Y25	Y28	Y12	Y08	Y14	Y02	Y13	Y21	Y03	Y27
Average (HB10/3000)	→	308	314,6	307,0	303	312	298,0	306	304,8	306	302,8	309,4	293	302	312,2

Point's number in the ellipse: 13
Total points number: 14
[(1)] : Laboratory excluded of the calculation with 95% confidence.

Font: Oliveira (1990).

BRINELL TEST		***Sample X***	***Sample Y***
Total average	→	*348,9*	*306,1*
Standard deviation	→	*7,6*	*4,5*
Variability Coefficient	→	*2,2%*	*1,5%*

The other tables were hidden on propose, therefore they are only for illustration the data gathering.



Font: Oliveira (1990).

Figure 9. Ellipses with 95% confidence level for Brinell tests (left) and Vickers tests (right).

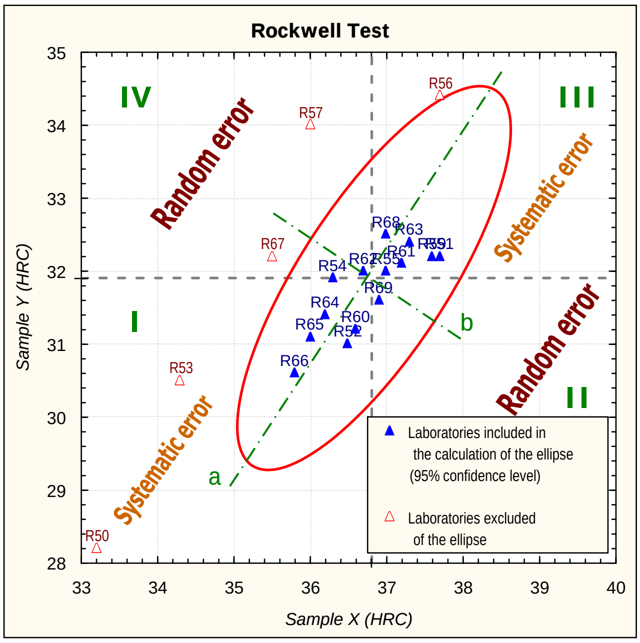


Figure 10. Ellipse with 95% confidence level for Rockwell tests, illustrating the error types.

2.3. Final Considerations

Only starting from the content of the information to be obtained in this study, it will be larger possible conclusions, that should be strongly marked by the variability analysis of the laboratories results and your influence on metrological reliability of information generated in tests, besides, evaluating the compatibility between the several results of the tests performed by MTLs, and also, a possible influence of sample measurements results.

With the return of the filled out questionnaires, the participants' profile will be drawn and it will be made a check-list of norms requisites, it can investigate the correlation between the technical (PI) and management performance (requisites of norm ABNT NBR ISO/IEC 17025, 2001), and also, to verify the current technician-administrative structure is satisfactory and competitive internationally. As other examples of PT applications, is to look for obtaining and development reference materials, evaluation of accuracy and possible tendencies and profile of reproducibility.

Some considerations on the Youden Methodology, mainly in relation to construction of the confidence ellipse, they are possible, analyzing Figs. 9 and 10. The ellipse was built in such a way that the probability of a random point (X, Y), corresponding to a MTL, be contained inside of her, be of 95%. Therefore, MTLs that were out of the ellipses should detect and to correct the causes of errors in perform of tests, when they are already with the final report of the PI.

The center of the ellipse is determined by intersection of corresponding straight line the averages for each one of the samples, being considered the results obtained by the laboratories that were not eliminated of the calculation of the ellipse. It is formed, four quadrants, us which the points are distributed in agreement with the averages of each MTL (Fig. 10). Moved away data of crossing the central lines and drawings in the quadrants II and IV represent casual or random errors, indicating that a worse performance might have happened in one of the samples (high variability in MTL). Moved away data of crossing the central lines and drawings in the quadrants I and III represent systematic errors of laboratory (modification method; equipment calibration). Closer to straight line that goes by quadrants I and III, and closer of the crossing of the lines central, more solid it was the result of laboratory.

3. Acknowledgements

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