# A proposed method using t-test for stability check in proficiency testing in tensile and hardness tests

Riham. S.Hegazy[1]* and Magdy I. Mohamed[1]

[1] force and material metrology Lab., National Institute of Standards (NIS)
* Corresponding author email: Riham.hegazy@nis.sci.eg

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Abstract

Proficiency testing is a very powerful tool for laboratories to continuously monitor the validity of the results, and is considered one of the technical requirements for accreditation according to ISO 17025. Stability check for the test samples or artefacts is important to ensure that these samples are fit for purpose, and give consistent performance over a specified period of time. The aim of this work is to propose a method for stability check using t-test as an alternative method of that followed in ISO 13528. The proposed t-test method is applied on proficiency test for Rockwell hardness test, Brinell hardness test, and tensile test for both PVC (Polyvinyl Chloride), and steel reinforcement bars. The results of stability check are analyzed, and compared with the results obtained from the convenient method of ISO 13528. The proposed method excelled the convenient method followed by ISO 13528. The main advantage of the proposed method using the t-test is that it is independent of the participants' results; it depends only on the results from the lab, which performs the stability check

*Keywords*: Proficiency test, Stability check, t-test, Hardness test and Tensile test.

## 1 Introduction

Proficiency testing is concerned with the determination of laboratory performance by means of interlaboratory comparisons. Proficiency testing determines the performance of individual laboratories for specific tests or measurements and is used to monitor laboratories performance. The involved laboratory undergoes practical tests and/or calibrations, and then the results are compared with the corresponding results of the other laboratories. In addition to the importance of proficiency testing as one of the requirements for the accreditation of tests and calibration laboratories according to ISO 17025 [1], it has many benefits in verifying the competence of the personnel carrying out measurements, the suitability of the measurement methods, procedures used, and the accuracy of the measuring instruments, as well as environmental conditions in the laboratories. In a proficiency test, one or more artefacts are circulated among a number of laboratories. Each laboratory performs its measurements (tests or calibrates the artefacts) according to a predefined set of instructions and then reports its results to the proficiency test provider. Each laboratory's results are compared to the assigned value of the employed artefact [2]. The statistical design of a PT scheme must be appropriate. The method of data analysis should be chosen so as to accurately explain the diversity in results amongst participating laboratories [3,4]. Since the proficiency test takes some time to rotate the artefacts or samples between the participating laboratories, it is necessary to check the stability of the artefacts and/or specimens over this period of time, as in ISO 17043 [5]. To determine whether the samples undergo any significant change during the proficiency test, stability testing should be carried out [6, 7]. ISO 13528 [8] gives a convenient model for testing stability in proficiency tests. This study proposed the using of t-test analysis for the stability check the test items as an alternative method to that followed in ISO 13528 [8]. The proposed analysis will be carried out for both tensile and hardness specimens. A comparison between the results obtained by the two methods is presented.

## 2 Theoretical Background

According to the preliminary approach [9], in this study, the variances of two means for two normal distributions are unknown but equal.
Suppose that $x_1$, $x_2$, $x_3$, ………$x_{nx}$, are independent, normally distributed repetitions taken by the pivot laboratory before a proficiency testing round with mean $\mu_x$ and variance $\sigma^2$.
Similarly, $y_1$, $y_2$, $y_3$, ………$y_{ny}$, are independent, normally distributed repetitions taken by the pivot laboratory after a proficiency testing round with mean $\mu_y$ and variance $\sigma^2$.
By combining properties of these two normal distributions, $\bar{x} - \bar{y}$is normally distributed with mean $\mu_x - \mu_y$and variance $\sigma^2 \left(\frac{1}{n_x} + \frac{1}{n_y}\right)$. Therefore, the term: has a z distribution and the term $\frac{(\overline{x} - \overline{y}) - (\mu_x - \mu_y)}{\sigma\sqrt{1/n_x + 1/n_y}}$ $\frac{(n_y-1)s_y^2}{\sigma^2} + \frac{(n_x-1)s_x^2}{\sigma^2}$ has a $\chi^2$ distribution with ($n_x$+$n_y^{-2}$) degrees of freedom.
Where $n_x$ ,$n_y$ is the number of two repetitions x,y respectively
By the definition of t distribution, the term $t_2$ is defined as :

$$t_2 = \frac{(\bar{x}-\bar{y})-(\mu_x-\mu_y)}{\sqrt{\frac{(n_x-1)s_x^2+(n_y-1)s_y^2}{n_x+n_y-2}}\sqrt{\frac{1}{n_x}+\frac{1}{n_y}}} \quad (1)$$

So, $t_2$ has a t distribution with degree of freedom ν equals $n_x$+$n_y^{-2}$. The following test can be used to determine whether the difference between the averages of the repetitions taken by the pivot laboratory before and after a proficiency testing round is significant to check the stability of the artefact:
If $|t_2| > t_{\alpha/2\,;\,v}$ then $\bar{x}$is significantly different from $\bar{y}$for 1-α confidence. The value of $t_2$ can be determined from equation 1 with assuming $\mu_x - \mu_y$=0, the value of $t_{\alpha/2}$ can be determined from the student distribution table. The means and standard deviations for x and y are calculated from the following equations:

$$\bar{x} = \sum_{i=1}^{n_x} \frac{x_i}{n_x} \quad (2)$$

$$s_x = \sum_{i=1}^{n_x} \frac{(x_i - \bar{x})^2}{n_x - 1} \quad (3)$$

$$\bar{y} = \sum_{i=1}^{n_y} \frac{y_i}{n_y} \quad (4)$$

$$s_y = \sum_{i=1}^{n_y} \frac{(y_i - \bar{y})^2}{n_y - 1} \quad (5)$$

The following section shows a numerical example for the proposed method.

## 3 Numerical Examples

This section discuses using t-test for analysing the stability check in three different proficiency tests with 95% confidence level. The results are compared with those evaluated by the method mentioned in clause B.5 in ISO 13528 [8]. The three proficiency tests are: hardness test, tensile test for PMMA, and tensile test for steel reinforcement bars. The inequality B.17 in ISO13528 [8] is used to check the stability of the samples. Then the proposed t- test method is compared with it:

$$|\bar{x} - \bar{y}| \leq 0.3\sigma_{pt} \quad (6)$$

Where $\sigma_{pt}$ is the standard deviation for proficiency test assessment.

### *3.1 Hardness Test*

The aim of this proficiency testing (PT) scheme is to give the participants an opportunity to demonstrate their proficiency in "Rockwell, and Brinell Hardness Test.” participants will demonstrate technical competence for the measurement of parameters. A Rockwell and Brinell Hardness Blocks have been used in this PT scheme. To check the stability of the hardness blocks, the two methods are applied, and the results are shown in table 1. The test was done according to ISO 6506-1 for Brinell hardness test and ISO 6508-1 for Rockwell hardness test. The two tests were done at a temperature of 23°C.

#### *3.1.1 Rockwell hardness*

The Rockwell hardness test method is based on indenting the surface with a conical diamond indenter. The test force was applied to the test piece by the conical indenter without shock or vibration. The preliminary force (10 kg) was applied for 12 sec. The initial indentation depth was measured, then an additional force F1 (150 kg) was applied in 5 seconds, and maintained for 10 sec. The additional test force, F1 was removed, while the preliminary test force, F0, was maintained. The final reading of the indentation depth was measured. The Rockwell hardness number was calculated from the permanent indentation depth, h (the difference between the final and initial indentation depth), using the formula

$$\text{Rockwell hardness} = N - \frac{h}{s} \quad (7)$$

Where N, S are two constants, N=100 and s=0.002 according to ISO 6508-1

#### *3.1.2 Brinell harness*

The test force of the Binell hardness test was 187.5 kgf. In order to test the largest representative area of the test piece, 2.5 mm ball indenter was chosen. The test piece was placed on a rigid support to prevent movement during the test. The ball indenter was forced into the test surface by a force in a direction perpendicular to the surface, without shock or vibration, until the applied force attained 187.5 kgf. The time from the initial application of force to the full test force was 5 sec. The duration of the test force was 10 sec. The diameter of each indentation was measured in two directions perpendicular to each other. The arithmetic mean of the two readings was used for the calculation of the Brinell hardness from the formula

$$\text{Brinell hardness} = 0.102 \times \frac{2F}{\pi D(D-\sqrt{D^2-d^2})} \quad (8)$$

**Table 1:** *Stability check for hardness proficiency testing scheme.*

| Reading No. | HRC Sample No. 1 | | HRC Sample No. 2 | | HB (187.5/2.5) Sample No. 3 | |
|---|---|---|---|---|---|---|
| | **Before PT round** | **After PT round** | **Before PT round** | **After PT round** | **Before PT round** | **After PT round** |
| 1 | 64.5 | 64.0 | 50.0 | 50.0 | 229.0 | 226.6 |
| 2 | 64.0 | 64.0 | 50.0 | 50.0 | 230.0 | 225.9 |
| 3 | 64.5 | 64.5 | 50.5 | 50.0 | 228.0 | 226.2 |
| 4 | 64.0 | 64.0 | 50.5 | 50.5 | 229.0 | 227.5 |
| 5 | 63.5 | 63.5 | 50.5 | 50.5 | 228.0 | 227.8 |
| $\bar{x}$ | 64.1 | | 50.3 | | 228.8 | |
| $s_x$ | 0.418 | | 0.274 | | 0.837 | |
| $n_x$ | 5 | | 5 | | 5 | |
| $\bar{y}$ | 64.0 | | 50.2 | | 226.8 | |
| $s_y$ | 0.354 | | 0.274 | | 0.822 | |
| $n_y$ | 5 | | 5 | | 5 | |
| $\nu$ | 8 | | 8 | | 8 | |
| $\|t_2\|$ | 0.408 | | 0.577 | | 3.814 | |
| $t_{\alpha/2,\nu}$ | 2.306 | | 2.306 | | 2.306 | |
| $\|\bar{x}-\bar{y}\|$ | 0.1 | | 0.1 | | 2 | |
| $0.3\sigma_{pt}$ | 0.549 | | 0.543 | | 1.157 | |
| Stability status | Fulfil | | Fulfil | | Not Fulfil | |

The mean of the results of the Rockwell hardness sample No1 before the beginning of the PT scheme was 64.1 HRC, and after the PT scheme was 64 HRC. The t test method indicates that $|t_2|$=0.408 < $t_{\alpha/2,\nu}$= 2.306,which means that there is no significant difference between the measurements before, and after the PT scheme (i.e. the sample is stable) Similarly, the hardness Rockwell Sample No. 2 $|t_2|$=0.577 < $t_{\alpha/2,\nu}$= 2.306, also There is no significant difference between measurements taken before and after the PT scheme, indicating that the sample is stable. For Brinell hardness sample, the mean of the measurements before the PT scheme was 228.8HBW, and after the PT scheme equal 226.8HBW. The t- test method indicates that

$|t_2|$=3.814 > $t_{\alpha/2,\nu}$= 2.306 which means that there is significant difference between two measurements (i.e. the sample is not stable during the PT scheme). The same results obtained by the Conventional method followed in ISO 13528, where the mean of the two Rockwell hardness sample No. 1 and 2 $|\bar{x} - \bar{y}|$=0.1 < $0.3\sigma_{pt}$ = 0.54 Which means that the two samples are stable during the PT scheme. For the Brinell hardness sample $|\bar{x} - \bar{y}|$=2 > $0.3\sigma_{pt}$ = 1.157 which means that the two samples are not stable during the PT scheme.

From the previous example, the stability of the two samples of Rockwell hardness is fulfilled by the two methods. For the Brinell hardness block, the two methods show that the hardness block is not stable.

### *3.2 PVC Tensile Test*

This proficiency testing (PT) scheme is designed to measure the competency of the participants in performing "Tensile Strength and Elongation for PVC (Polyvinyl Chloride) material (Before Aging and After Aging)". The tensile test and elongation were performed using dumb-bells shape samples and were carried out on samples before and after aging. The sample is shown in Fig 1. The aging of samples is carried out according to IEC-60811 (part 401). The specimens were suspended vertically and substantially in the middle of the oven so that each test piece is at 20 mm from any other test pieces. The test pieces were kept in the oven at the temperature of 100 °C for 7 days. As soon as the ageing period is completed, the test pieces were removed from the oven and left at ambient temperature for 16 h, avoiding direct sunlight. Tensile test was performed at ambient temperature of 23°C. The test pieces were held in such a way that the force is applied as axially as possible, in order to minimize bending. The test rate was 50 mm/min until fracture. The tensile stress was calculated by dividing the maximum force by the original cross-sectional area of the specimen.

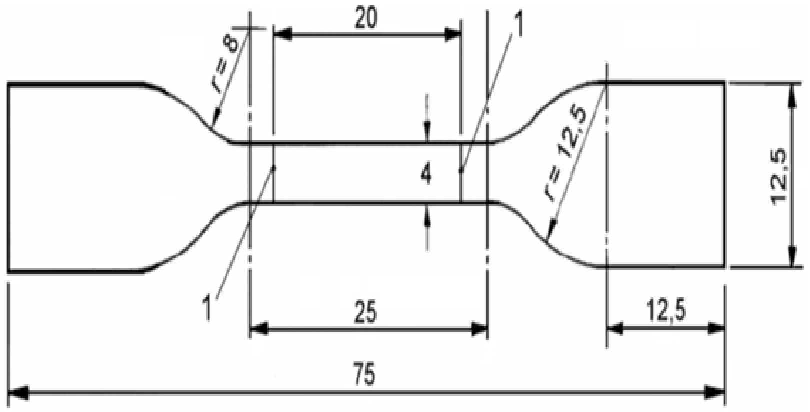


**Figure 1:** The dumb-bell shape sample

In order to determine the elongation after fracture, the two broken pieces were carefully fitted back together so that their axes in a straight line. The final length was measured. The percentage elongation after fracture was calculated, from the form

$$\text{Elongation}=\frac{Lf-Lo}{L0}\times 100 \qquad (9)$$

Where Lf is the final length after fracture, Lo is the original gauge length. Five samples are tested for checking the stability as shown in table 2.

**Table** 2: *Stability check for PVC samples.*

| Sample No. | Before Aging Tensile Strength (MPa) | | Elongation (%) | | After Aging Tensile Strength (MPa) | | Elongation (%) | |
|---|---|---|---|---|---|---|---|---|
| | **Before PT round** | **After PT round** | **Before PT round** | **After PT round** | **Before PT round** | **After PT round** | **Before PT round** | **After PT round** |
| 1 | 15.31 | 15.64 | 292.00 | 272.55 | 15.43 | 15.78 | 272 | 266 |
| 2 | 13.46 | 15.83 | 296.40 | 282.02 | 15.62 | 15.10 | 284 | 296 |
| 3 | 15.45 | 16.62 | 290.32 | 290.95 | 16.30 | 14.40 | 277 | 250 |
| 4 | 15.76 | 15.76 | 307.00 | 308.00 | 16.80 | 18.28 | 280 | 304 |
| 5 | 16.34 | 16.93 | 302.00 | 320.00 | 17.50 | 15.04 | 277 | 260 |
| $\bar{x}$ | | 15.3 | | 297.5 | | 16.3 | | 278.0 |
| $s_x$ | | 1.083 | | 6.955 | | 0.852 | | 4.416 |
| $n_x$ | | 5 | | 5 | | 5 | | 5 |
| $\bar{y}$ | | 16.2 | | 294.7 | | 15.7 | | 275.2 |
| $s_y$ | | 0.580 | | 19.251 | | 1.512 | | 23.520 |
| $n_y$ | | 5 | | 5 | | 5 | | 5 |
| $\nu$ | | 8 | | 8 | | 8 | | 8 |
| $\lvert t_2\rvert$ | | 1.623 | | 0.310 | | 0.786 | | 0.262 |
| $t_{\alpha/2,\nu}$ | | 2.306 | | 2.306 | | 2.306 | | 2.306 |
| $\lvert\bar{x}-\bar{y}\rvert$ | | 0.9 | | 2.8 | | 0.6 | | 2.8 |
| $0.3\sigma_{pt}$ | | 1.17 | | 19.251 | | 1.512 | | 23.52 |
| Stability status | | Fulfil | | Fulfil | | Fulfil | | Fulfil |

According to the t- test method, the tensile strength results of the PVC sample before aging and before the Beginning of the PT scheme $|t_2|$=1.623 < $t_{\alpha/2,\nu}$= 2.306, this means that there is no significant difference between the measurements before, and after the PT scheme (i.e. the samples are stable). Similarly, for the elongation $|t_2|$=0.310 < $t_{\alpha/2,\nu}$= 2.306, there is no significant difference between the measurements before, and after the PT scheme, which means stable sample. For the samples after ageing regarding the tensile strength $|t_2|$=0.786 < $t_{\alpha/2,\nu}$= 2.306 which means that there is no significant difference between two measurements , ( the sample is stable during the PT scheme), the same for elongation $|t_2|$=0.262 < $t_{\alpha/2,\nu}$= 2.306 .The same results were obtained by the conventional method followed in ISO 13528.The mean of the two Rockwell hardness sample No. 1 and 2 $|\bar{x}-\bar{y}|$ < $0.3\sigma_{pt}$ for all measurements , which means that the two samples are stable during the PT scheme for tensile strength and elongation.It was shown in the previous example that the two methods for stability check leads to the same results. The proposed method using t-test agrees well with the conventional method for ISO 13528, the stability of the samples was fulfilled (before, and after aging).

### *3.3 Tensile Test for Steel Reinforcement Bars*

The aim of this proficiency testing (PT) scheme is to give participants an opportunity to demonstrate their proficiency in "Tensile Test for Steel Reinforcement Bars”

Steel reinforcement Test Bars are used in this PT scheme with a 12 mm nominal diameter, 300 mm length, and 60 mm gauge length. Each participant receives five test samples. Nine samples were tested for stability check. The results for stability check are shown in table 3. The test was done in accordance with ISO 6892-1. The test was done at 23°C.

The test pieces were gripped by wedges to maintain the force in axial direction to minimize bending. In order to ensure the alignment of the test piece and grip arrangement, a preliminary force was applied with a value of 5 % of the specified or expected yield strength. The test rate was 10 mm/min Yield stress was determined from the force-extension curve, and was determined from the lowest value of stress during plastic yielding, ignoring any initial transient effects. The value was calculated by dividing this force by the original cross-sectional area of the test piece. The ultimate tensile strength was calculated by dividing the maximum force obtained by the original cross section area of the test piece.

**Table 3:** *stability check for steel reinforcement bars*

| Sample Code | Yield Stress (MPa) | | Ultimate Tensile Strength (MPa) | |
|---|---|---|---|---|
| | Before PT round | After PT round | Before PT round | After PT round |
| 1/A | 654.31 | 655.20 | 745.32 | 764.36 |
| 2/A | 650.20 | 658.29 | 743.91 | 741.14 |
| 3/A | 652.12 | 653.26 | 750.31 | 724.5 |
| 1/B | 660.71 | 644.02 | 745.91 | 725.82 |
| 2/B | 669.20 | 682.11 | 744.51 | 758.2 |
| 3/B | 651.70 | 659.87 | 748.67 | 746.91 |
| 1/C | 670.54 | 638.19 | 741.23 | 725.76 |
| 2/C | 632.12 | 675.98 | 752.95 | 760.07 |
| 3/C | 640.43 | 664.67 | 746.23 | 747.71 |
| $\bar{x}$ | 653.5 | | 746.6 | |
| $s_x$ | 12.417 | | 3.554 | |
| $n_x$ | 9 | | 9 | |
| $\bar{y}$ | 659.1 | | 743.8 | |
| $s_y$ | 13.967 | | 15.605 | |
| $n_y$ | 9 | | 9 | |
| $\nu$ | 16 | | 16 | |
| $\lvert t_2 \rvert$ | 0.896 | | 0.512 | |
| $t_{\alpha/2,\nu}$ | 2.12 | | 2.12 | |

| | | |
|---|---|---|
| $\lvert\bar{x}-\bar{y}\rvert$ | 5.6 | 2.8 |
| $0.3\sigma_{pt}$ | 6.85 | 7.6 |
| **Stability status** | Fulfil | Fulfill |

## 4 Conclusion

Proficiency testing is the most powerful tool used to measure the competency of the participants to do specific test or measurements. Additionally, it is important to evaluate the continuous performance of the laboratories. Stability check of the samples or artifacts is a main requirement in ISO 17043 for PT providers to ensure that the samples or artifacts used in the PT schemes give the same performance over a specified period of time. ISO 13528 present a convenient method for checking the stability of samples, or artifacts. This study presents an alternative method using t-test. The proposed method was applied in three PT schemes (Rockwell hardness test, Brinell hardness test, tensile test for PVC, and tensile test for steel reinforcement bars). The results show that the proposed method agrees well with the method of ISO 13528, otherwise, this proposed method excels over the convenient method followed by ISO 13528. The main advantage of the proposed method using t- test is that it is independent of the participants' results, it depends only on the results from one lab, which perform the stability check.

## 5 Declarations

### *5.1 Study Limitations*

None.

### *5.2 Funding source*

None.

### *5.3 Competing Interests*

The authors have no financial or proprietary interests in any material discussed in this article.

### *5.4 Ethical Approval*

Not Required

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