TECHNICAL BULLETIN: DESCRIPTION AND ADVANTAGES OF INTERLABORATORY STUDIES

1 Introduction

Interlaboratories are studies in which more than one laboratory is involved[6.3] and, in general terms, are used through two main approaches: to valorize the "measurement systems" of laboratories (see section 2) or to evaluate the accordance of the established accuracy in a test method. For this reason, interlaboratories may have different specific uses[6,7]: characterization of a method (**Accuracy study**) certification of a material (**Certification study**) and the evaluation of performance of a laboratory compared to other similar (**Proficiency test**). In these types of interlaboratories, the number of participants should ideally be greater than six (6)[6.4].

At the same time, there are statistical comparisons between two or more laboratories, which are based on hypothesis tests associated to the correspondence of results from the same test method (Interlaboratories and Crosschecks), called Round Robins.

Round robins are extremely useful for the continuous and periodic monitoring of the performance of a laboratory (for plant quality control and research), where confidence intervals are also calculated and compared to detect trends that allow to identify improvement opportunities to participants. These types of studies have the advantage of being an effective tool to maintain and improve the competence of a laboratory.

In this bulletin, only Round Robins studies are described.

A successful interlaboratory is based on a good organization; by choosing the property to be evaluated which is preferably to be useful in the market; by selecting the participants, specimens and test levels to be included[1] and finally the number of replicates.

2 Most probable sources of Variability in Measurement Systems

A measurement system (analyst-team-sample-environment) can be affected by random errors (no control on these ones) and systematic errors (associated with failures that can be corrected). The variation sources may occur by several reasons that must be taken into account before any type of comparison between laboratories. These kinds of sources include:

1. Traceability of the measuring equipment to national or international standards.
2. Test samples.
3. Measuring equipment.
4. The analyst and the procedure (see example in Figure 1).
5. Environmental conditions.
6. The test run time (especially required for interlaboratory methods associated with precision studies).

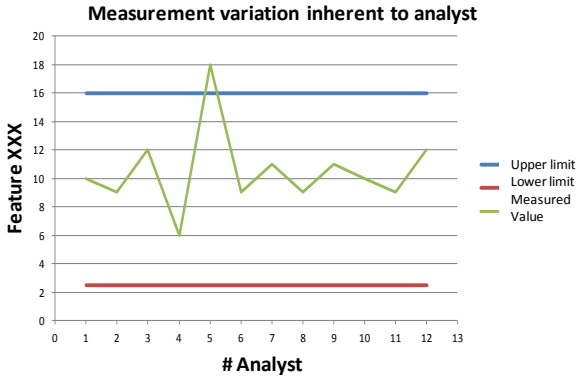

Figure 1. Example of possible variability inherent to the analyst, on the same test method

An interlaboratory study has the advantage of showing to participants the influence that each one of these sources can have on their data.

The data statistically evaluated on interlaboratory study can vary according to the type of study; for example, Round Robins work with the number of measurements or "test replication[2]" (each of these

[1] Test level: different response ranges of the same property.

[2] Test replication: exact reproduction of the test, on similar samples and in with shortest possible time between each one.

tests, with sample replicate[3] established in the corresponding regulations).

3 Advantages of Interlaboratory Studies

Analytical laboratories in a manufacturing company must provide reliable information of the raw material status, material in the processing stage and products, to allow the plant to operate within a window of capacity, quality and costs that will maintain the profitability and competitiveness of the business, as well as the confidence of the customers in the products and brand that this company sells.

In cases where a product or raw material is wrongly rejected or a defective product is used , it would indirectly result in a decrease of operating capacity of the plant and / or will be an increase in its operating costs, because in some further time would be operating with problems, waste generation (from products and / or raw materials), return of material which will require to be reprocessed, and also will demand a larger consumption of raw materials or energy (steam, electricity).

Also, customers will lose satisfaction and they will be tempted to migrate to other suppliers of product/raw material with a better-performance.

With the improvements introduced by interlaboratories studies, participants (who may become upstream or downstream of a process) could reduce (monetary) losses in their production as they achieve greater certainty in their test results, which are in charge of approving or failing a product or raw material.

In this regard, interlaboratories have many advantages that allow to participants to work for continuous improvement goal of their procedures, some of these are:

- They are useful for periodic monitoring of the operation and authenticity of a plant quality control laboratory, so they are an effective tool to maintain and improve the competition of the quality control.
- Because of interlaboratories are focused on the process, they allow a total control over each one of the procedures that include the tests, since they show to the participants the influence of most of the sources of variability on the data (see section 2)
- They improve the quality control of their plants.
- If there are specifications of the material used, these will be checked for their fulfillment or compliance.
- Cross-checks and interlaboratories are generally referenced to some national or international regulations. When the requirements of a standard are fulfilled (such as ASTM E1601, for example), laboratories will increasingly have more competent operators that will be familiar with their testing methods.
- Interlaboratories guarantee to be unbiased comparisons, when participants follow what is established in the technical protocol.
- Interlaboratories check if the samples come from populations (from products or raw materials) with the same variance, by constructing a comparative variability analysis.

3.1 Other features[6.13]

Another main importance of an interlaboratory study is that it seeks to maintain a good performance of the laboratory for an extended period of time, and its results will be the reference of the good performance for later uses.

An unsatisfactory result of an interlaboratory study may be caused by a simple error, which inevitably can occur in all laboratories some time, but that does not mean that there is no competence to carry out the quality control. Precisely, this type of tests

[3] Sample replication: quantity of specimens tested in accordance to standards.

allow to users to identify errors and to introduce the corresponding corrections.

4 Parts of a Round Robin Interlaboratory [6.12]

The working group or "panel" in charge of planning the Round Robin interlaboratory, must select the number of participating laboratories, the material involved, the property to be evaluated and the type of study or statistical analysis that will be applied. Each participant must select the skilled analyst in the test methods and their applications, before carrying out the determinations[6.8]. The following sections show, in chronological order, the steps to be followed:

4.1 Selection of laboratories

Any laboratory that is considered qualified to carry out the selected test should have the opportunity to participate in the interlaboratory study, as long as there are equivalence of test methods[6.7]. There are different opinions about this selection:

According to ASTM E691[6.5], which aims to establish the accuracy of a test method, the personnel organizing the interlaboratory should evaluate the possibility of the laboratory to participate, depending on the existence of the operational test equipments available, the provision of qualified operators familiar with the test method, and the existence of sufficient time and interest showed by the analysts.

This ASTM Standard recommends as an optimal quorum, the participation of 30 laboratories, and also establishes a minimum of 6 laboratories to validate the analysis of the results.

On the other hand, according to ISO 5725[6.1], which establishes the truthfulness and precision of a test method, laboratories should not be exclusively those who have gained experience during the standardization of the method, but the group must contain laboratories where one of these acts as a "reference laboratory", to be able to demonstrate the accuracy for which the method can be carried out by experts.

In order to minimize the risk of any bias, it is important to include all laboratories that regularly use the method. The Standard requires calculating the optimal number of participating laboratories to minimize uncertainty in determining accuracy, but also indicates that the most common range for acceptable interlaboratories is between 8 and 15 laboratories.

4.2 Selection of the method to be evaluated[6.4]

The method includes the following aspects:

- Detection of the needs of the interlaboratory study, depending on the usefulness of the method for the participants.
- Availability of required test material.
- Skill, experience and willingness by analyst staff.
- A review of the regulations of analysis by the responsible of each laboratory, to verify that their method complies with the requirements.

The beginning of the interlaboratory is carried out once a consensus is reached by all the participants.

4.3 Selection of materials to be included in the interlaboratory

Test levels

The materials to be used in the interlaboratory study should represent those that are commonly evaluated with the test method and, if possible to include several materials. It is expected that those materials show extreme values of the property to be measured (denominated A, B, C and D on Figure 2). The levels to include are defined as follows:

- The different kind of materials to be evaluated.
- The difficulty and costs associated to obtaining, processing and distributing samples.
- The difficulty and time invested to carry out all test.
- In the case that tests are necessary as a commercial and / or legal requirement, the purpose of the test results should be included for any participant.

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In the interlaboratory organized by Polintera reference material is commonly used, whose basic properties (usually melt flow index and density) have been determined as homogeneous and stable according to ISO 5725. This determination allows discarding the natural variability of the material as the cause of the discrepancies in the measurements between different laboratories.

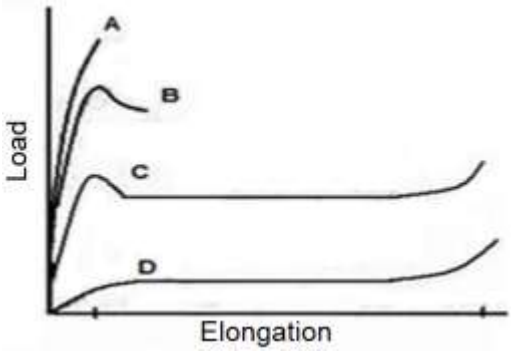

Figure 2. Different test levels (in this case, tensile strength)

4.4 Writing the Technical Protocol

This protocol is a document where the responsible of organizing the interlaboratory describes clear instructions of the experiment that will be carried out. The protocol has the following points:

- Project scope: includes the number of laboratories involved, the number and total description of the samples, and the test methods to be included (with the related standard).
- Type of Study: in this case a summary of the interlaboratory is described, the format that will have the result report of each participant and the type of statistical analysis to be applied (comparison of means test, multiple comparisons of means test, etc.).
- Design Of Experiments (DOE)[6.2]: this could vary according to the type of interlaboratory. The DOE includes the number of test replicates to be requested for the comparison; generally it is expected to use at least fifteen (15) data points. Also DOE includes any other detailed descriptions of the samples and how the evaluations will be conducted. See example of a DOE in Figure 3.

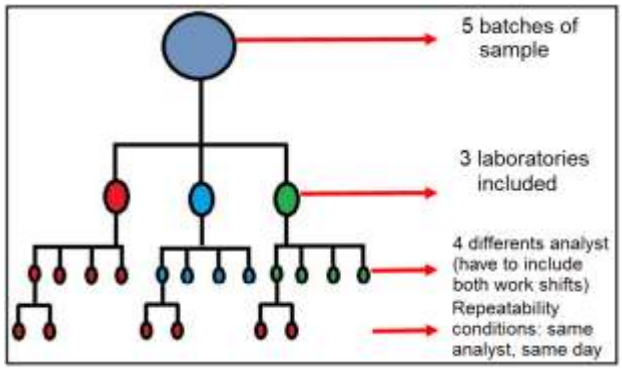

Figure 3. Example of a DOE

It is highly recommended to keep the design as simple as possible in order to obtain estimates of interlaboratory variability, without secondary effects.

- Contacts: this section describes all possible communication modes among the coordinator and the participants.
- Report: this document contains all results of the study and it should also be adapted to the type of study, and the possibilities of each participant.

Technical protocol must be reviewed by the participants, in order to reach an agreement about rules of the study, before starting.

4.5 Samples delivery

Prior to dispatching of samples, they should be randomly chosen within the original population, by using any method of arrangement, as is indicated in Figure 4. Occasionally, a third entity is responsible for performing the samples selection.

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Figure 4. Samples arrangement

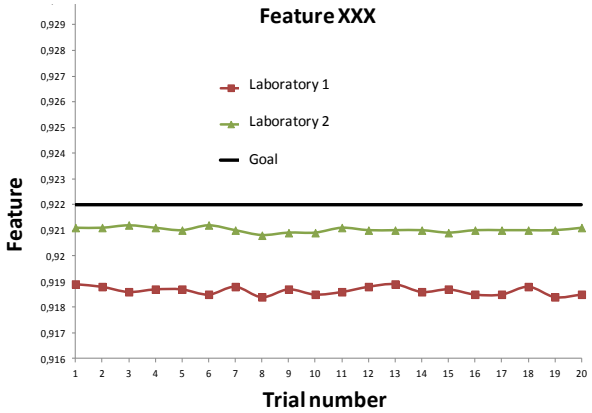

Figure 5. Graph comparison example.

All specimens must be packed to prevent any damage, like impact, overload or heating, and depending on the type of sample to be evaluated, the package should protect the stability and the material characteristics during transportation.

4.6 Time frame for carrying out tests

In a work schedule, must be established a deadline for receiving data, in order to set guidelines for the participants and ensure the highest equivalence among time frames.

4.7 Statistical analysis and variability study

In this case, the parts that comprise this analysis, which represents main part of the interlaboratory comparisons, are broken down in order to provide to the reader the technical support for identifying the trends that they would present their results.

4.7.1 Graphic Comparison.

In order to make easier the understanding of all participants, the averages of the measurements are usually placed first to visually contrast them, in order to identify patterns or trends, which may incur subsequent statistical differences (see example on Figure 5).

4.7.2 Variance analysis[6.9] and confidence interval

In this case, statistical techniques to compare averages of the results of each laboratory are necessary, such as Analysis of Variance (or ANOVA), Hypothesis Testing study for Means, Multiple Comparisons of Means, comparison of Confidence Intervals and other tools.

This type of analysis makes possible to establish whether the variability obtained is due only to the measurements, for example by normal error of the test or measurement process, in which case it is possible to say that there are no statistically significant among averages of the participating laboratories.

By contrast, if the variability can be attributed to the execution of test in one or more of the participating laboratories, it indicates that there are statistically significant differences among the set of participating laboratories.

The confidence intervals for the average, for example, allow to have a quick visual comparison between averages to verify equivalence of the participating laboratories, since they represent the range of values where there is a certain probability (level of confidence) to have the true value of the average (example in Figure 6).

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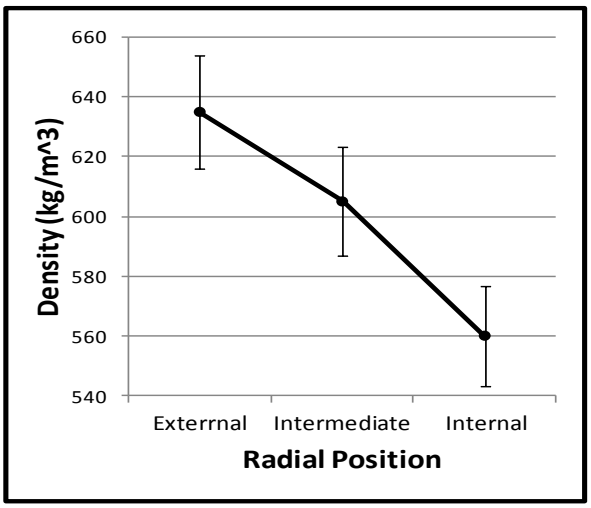

Figure 6. Example Confidence Intervals for the Average of a material property

Another technique is the Multiple comparison of Means, that is designed to collate two or more reported samples, and for verifying whether they present or not statistically significant differences, through the Least Significant Difference (LSD) test.

In some scenarios where there are more than two laboratories, this technique allows to compare different pairs of data, making an automatic permutation of the laboratories and, when identifying pairs that can be considered equivalent, it allows to determine a consistency ratio (normally in %) of the results of the interlaboratory.

$$\text{Consistency ratio} = \frac{\#\text{ of consistents pairs}}{\text{total number of combinations}} \text{x}100$$

In each pair of laboratories evaluated it is also possible to apply the Null Hypothesis test to verify these statements.

4.8 Submitting results

Once the analysis is completed, the organizer submits the results to each participant according to the stipulated in the technical protocol. Usually, each laboratory receives information on its particular performance that later it is compared against all the information available from the rest of the laboratories by using averages and ranges, in order to maintain the confidentiality of the same.

These reports help to define corrective and preventive actions if significant deviations were identified. It can also be used to show evidence of compliance with clients and auditors.

5 Conclusions

Interlaboratory studies are an efficient tool that allows analyzing the skills of analytical laboratories, for productive companies and research centers, through the evaluation of the agreed properties, made in a systematic and rigorous way.

Interlaboratory results allow to reaffirm the ability to perform trials and to determine corrective and preventive actions, if necessary.

6 References.

6.1 **ISO International Organization for Standarization.** ISO Standard 5725:1994(E), TC69 SC6. "Accuracy (trueness and precision) of measurement methods and results", part 3.

6.2 **ISO International Organization for Standarization.** ISO Standar 3534, TC69 SC1 "Vocabulary and Symbols" (parts 1 to 6).

6.3 **International union of pure and applied chemistry. IUPAC**. Nomenclature of interlaboratory analytical studies.

6.4 **ASTM E1601:98** "Standard Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method". ASTM International. West Conshohocken, PA, 2003. DOI: 10.1520/E1601-98. www.astm.org.

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