Bipea Partenaire de votre Qualité

Contact - Best Wishes for 2008

36th Year Letter n°100

Editorial

Today Bipea Newsletter is celebrating its 100th issue! The advantage of round numbers is that they conjure up others. This 100th issue makes me think of our 1,000 members spread over 50 countries. And to serve our 1,000 members, we ship more than 7,000 samples and handle 70,000 statistical data every month.

Bipea is a non-profit association which is called in French "Association under the French Law of 1901". Here is another figure that recalls Bipea's fundamentals. Bipea is an "association" and therefore it serves its "members." This concept goes well beyond the sales relationship, anchoring the relationship between the terms "efficiency" and "partnership."

The Board of Directors and its Chairman, the TBM and its Chairman, the Commission Chairmen and participants as well as the 17 permanent Bipea staff work hand-in-hand to meet members' expectations and to accomplish the missions established when Bipea was created in 1970.

As Bipea's new Director, I want to perpetuate the partnerships we've developed for nearly four decades with the multiple joint trade organizations present in our governing bodies. These partnerships have enabled Bipea to become a reference is several areas. I want to increase Bipea's involvement in standardization to support development of the professions in Bipea. I believe that Bipea's know-how should support the branches in their standardization initiatives. Bipea should also develop a strong relationship with its members. This inevitably involves organizing internship, training courses, seminars and congresses (and even individual visits). I hope this objective can become a reality in 2008! We also have to stimulate participation in Bipea Commissions. Indeed, these meetings should be an opportunity to exchange with members and provide them with information. I would like to see increased preparatory work undertaken to support Commission Chairmen in their volunteer work.

With 40% non-French laboratories, Bipea is a multinational player! The Board of Directors wants to strengthen Bipea internationally. This essential development will reinforce the benefits of the schemes offered by Bipea and, why hide it, ensure diversified economic development. At Bipea the term "association" also rhymes with economic efficiency. We don't want to miss international growth opportunities. To succeed internationally, Bipea will invest in training its staff and will adapt its productive assets to its linguistic plurality.

I want to take advantage of this 100th issue of the Bipea Newsletter to wish you all a prosperous and successful 2008. In 2008, Bipea will remain your Quality partner.

Bruno BERKEN

Bipea Manager

Determining the assigned value in Bipea Proficiency Testing Schemes

In chapter 5, the NF ISO 13528:2005 standard describes five ways to determine the assigned value in proficiency testing by interlaboratory comparisons. Remember that in this context, the assigned value is a synonym of the "true value" as defined in the International Metrology Vocabulary; namely "§2.12. Value attributed to a magnitude by agreement for a given use." In Bipea's interlaboratory comparison reports, the assigned value is named by convention the "reference value."

The standard specifies that the organiser of the interlaboratory comparisons (OILC) is responsible for choosing between these five methods. As specified in the Cofrac reference guide (Lab Cil ref 02), the OILC can consult a technical group comprised of experts in the field or the participants in the comparison to confirm his choice.

It is recommended that participants should know the method chosen to determine the assigned value before the comparison, but the assigned value should not be disclosed as long as the results haven't been provided to the OILC.

In this text, the five determination methods are presented based on their order of presentation in the standard. Therefore they are classified according to whether the assigned value is determined or not based on the participants' proficiency testing results. The methods most often used by Bipea are shown in chapter 2 where the assigned value is determined based on the participants' results. For each test, this information is shown, criterion-by-criterion, in the interlaboratory comparison test report appendices in the table, "the specialised commission's reference and tolerance value."

1 – Determining other than with participants' proficiency testing results

1.1 - Formulation [see NF ISO 13528:2005, 5.2]

This approach can be used when the test material is prepared by mixing the components in the specified proportions or by adding a specified proportion of a

Content

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La lettre Contact du Bipea
Directeur de la publication : Bruno Berken
Rédaction : Gérard Roine
Bipea : 6 à 14, avenue Louis Roche
92230 GENNEVILLIERS
Tél. : +33 (0)1.47.33.54.60
Fax : +33 (0)1.40.86.92.59
E'Mail : Contact@bipea.org – Site : www.Bipea.org

Determining the assigned value in Bipea Proficiency Testing Schemes

component to a base matrix. This approach is appropriate when manufacturing samples individually. It is preferable to use another approach when the samples are prepared on a lot basis where the homogeneity has to be ensured.

However, use of this approach is limited because precautions have to be taken to ensure that:

- the basis material includes no traces of additive or the proportion of additive in the base material is known precisely
- the components are mixed homogeneously
- all sources of error are identified
- there is no interaction between the components and the matrix

The assigned value is not determined by analysis; rather it is assigned by construction or obtained by calculation. Therefore the assigned value's determination can be traced vis-à-vis the International System. When the proficiency testing is being validated, the assigned value must be compared to the robust mean of test participants.

This approach is rarely used in Bipea proficiency testing. The proficiency testing schemes concerned are scheme "23: Varietal identification of barley," scheme "19: Pesticides" and synthetic solutions for schemes "37: Feed water – Micropollutants" and "53: Waste Water – Micropollutants."

1.2 - Certified reference values [see NF ISO 13528:2005, 5.3]

A certified reference material (CRM) can be used as a test material. However, this approach has limits insofar as:

- it can be expensive to provide a CRM sample to each participant
- the list of available CRMs is limited
- identifying the CRM means participants know the assigned value
- the sample quantity may not be sufficient to test all of the requested criteria in the proficiency testing
- repackaging or dividing to perform a blind test may result in a change of the certified reference value

The certified value is then used as the assigned value. How the assigned value is determined can be traced.

No Bipea scheme is currently concerned by this approach. The OILC may resort to this approach when it sees a broad dispersion of results or there is an unexplained presence of two or more populations of results.

According to chapter 5.9 of the **NF EN ISO /CEI 17025:2005** standard, a laboratory can use a CRM as an individual approach to ensure its results are valid, the same as for proficiency testing.

1.3 - Reference values [see NF ISO 13528:2005, 5.4]

In this approach, the test material for the interlaboratory comparison must become a reference material (RM) before it is distributed to participants. To do this, the samples are subjected to tests with a CRM in a single laboratory using an appropriate method.

The constraints of constituting a RM include:

- the requirement to use a CRM that resembles the test material as closely as possible
- the requirement to analyse a large number of samples of future test RMs and CRMs under conditions of repeatability
- the choice of a laboratory to perform the analyses (accurate and reliable laboratory)

This method requires that there is no interaction between the materials used and the test conditions (stability, amount of the component in the test, etc.).

The assigned value, which is traceable, is deduced from a calibration based on the CRM's certified values.

As for the previous approach, no Bipea scheme is currently concerned. Even though this method saves the cost of distributing a CRM to all participants, it is not always possible to apply it because the list of available CRMs is limited. However, it can be implemented on a limited basis for the same reasons as those for using a CRM.

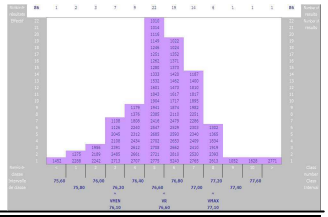
1.4 - Consensual values from non-participating expert laboratories [see NF ISO 13528:2005, 5.5]

In this approach, the test material is prepared for distribution to the participants. Some of these samples are then selected for analysis by a group of expert laboratories not participating in the interlaboratory comparison.

The assigned value is calculated as being the robust mean of the results provided by the group of expert laboratories, determined using the A algorithm in appendix C of the NF ISO 13528:2005 standard. When the comparison is being validated, the assigned value must be compared to the robust mean of test participants to identify a bias or drift.

Bipea doesn't use this approach very often because:

- it uses outside laboratories and therefore results in additional costs
- it requires controlling the expert laboratories with the same requirements as for a subcontractor
- generally enough expert laboratories participate in Bipea's interlaboratory comparisons. This approach



Determining the assigned value in Bipea Proficiency Testing Schemes

amounts to the case described in paragraph 2.1 of this article.

2 – Determination based on participants' proficiency testing results .

2.1 - Consensual values from participating expert laboratories [see NF ISO 13528:2005, 5.5]

This approach is identical to the one described above in paragraph 1.3 except that the expert laboratories participate in the interlaboratory comparison.

The test material samples are distributed to the participants. When the data are processed, the expert laboratories' results are identified and isolated in order to determine the assigned value.

A variation on this approach involves selecting among the participants so-called "control" laboratories. These laboratories are selected based on defined criteria, like:

- use of a standard
- use of a technique
- performance of laboratories selected based on previous tests (e.g.; accuracy and regularity)
- performance demonstrated by an accreditation
- the laboratory's use of a CRM in parallel with the test .

This list is not exhaustive because the technical group or the participants in the comparison can use other criteria (e.g.; recovery rate or verification of a calculation).

The assigned value is calculated as being the robust mean of the results of the group of expert or control laboratories, determined using the A algorithm. The assigned value must be compared to the robust mean of test participants to identify a bias or drift.

The variation of this approach has the advantage of:

- ensuring the robustness of the assigned value over time
- integrating new laboratories without influencing the assigned value, especially for schemes with few participants
- letting laboratories use other standards or methods than the recommended ones .

This variation is used in most of Bipea's testing schemes.

2.2 - Consensual values from participating laboratories [see NF ISO 13528:2005, 5.6]

The samples prepared from test materials are distributed to the participants.

The assigned value is the robust means of the results provided by all test participants.

The limits of this approach are, among others, as:

- Bias in estimating the assigned value due to the influence of the most frequent method or technique
- Lack of real consensus between participants (e.g.; regarding the use of a standard or method).

This approach is used in new Bipea proficiency schemes or in schemes when there isn't enough information to select a list of witness laboratories to determine the assigned value.

3. - Conclusion.

When developing the statistical plan for an interlaboratory comparison, the statistical model used must be documented to determine the assigned value and related data analysis techniques as well as to provide a description of the reasons for selecting them.

Among the five methods proposed in chapter 5 of the NF ISO 13528:2005 standard, the formulation method is not used very often in Bipea testing due to the constraints imposed to make samples. Likewise, it is rarely decided to use certified reference materials or reference materials due to their constraints of use in our food and environmental areas of activity.

The two methods used most often in Bipea testing are those that rely on test participants' results. The corollary of these two methods is therefore the use of robust statistical methods to determine the assigned value, such as the A algorithm .

Marie-Philippe Seiller,
Bipea Quality Manager

Validated by Mr. Max Feinberg,
Bipea Scientific Consultant

Bibliography :

- NF EN ISO 13528:2005 standard "Statistical methods used in proficiency testing by interlaboratory comparisons," Afnor, Paris
- COFRAC reference guide Lab Cil ref 02: Revision 02 - September 2007 "Organisers of interlaboratory comparisons – Accreditation requirements." Cofrac, Paris
- NF EN ISO/CEI 17025:2005 standard "General requirements concerning the competence of calibration and testing laboratories," Afnor, Paris

Study of the stability of samples

Julien Sarembaud, a PhD student in engineering at INAPG, has worked three years at Bipea on controlling the stability of samples, his thesis subject. Here he provides us his conclusions on this important subject .

Introduction

Among the various tools laboratories use to demonstrate and control the reliability of their results, a widespread approach involves participating in proficiency testing and/or using reference materials. Tests play a major role because they involve both the accuracy and traceability of measurements, to the point that their use is mandatory for laboratories accredited according to the ISO 17025 standard.

Reference materials

According to the ISO 30 guide, a reference material (RM) is defined as a material or substance whose property value(s) is (are) sufficiently homogeneous and well defined so it (they) can be used to calibrate an apparatus, assess a measurement method or reference values to materials.

Their increasingly frequent use by laboratories has led to an increase in the supply of these materials. The best known are certified reference materials (CRM) produced by international metrological institutes like the National Institute of Standards and Technology (NIST) or the Institute for Reference Materials and Measurements (IRMM). Substantial means and investments are required to prepare and certify them. The high prices and limited availability of these materials have stimulated the manufacture of new reference materials to meet these needs. The documentation booklet, AFNOR FD V03-115, considers the materials resulting from proficiency testing schemes (e.g.; those manufactured by Bipea) as external reference materials (ERM). However, in return, their production and sale assume that further information on supplied regarding their homogeneity and stability.

With a view to the use of these ERMs by laboratories, Bipea has implemented a procedure to check inview their homogeneity and should implement a similar procedure for their stability. One can distinguish:

- Short-term stability, which corresponds to proficiency testing schemes ranging from approximately two weeks to two months
- Long-term stability concerns the production of RMs used over long periods that can lost several years.

Stability studies

Stability studies are used primarily to determine a shelf life for all kinds of products, including RMs. Indeed food, chemical, biological and environmental products used as RMs are likely to change during their storage. These changes concern some of their parameters like the concentration of an analyte. If the change in these parameters results in a significant difference compared to their initial value, then the ERM no longer has the characteristics that defined it and can no longer be used by laboratories.

A product's shelf life based on these changes also depends on the legislative and commercial criteria. Depending on the product analysed, the modus operandi for stability studies and their interpretations vary.

However, irrespective of the type of product studied, there are two basic principles for these stability studies:

- Record the change in the elements and substances and even their disappearance
- Note the appearance of known degradation products

Long-term stability studies carried out in Bipea include several steps:

- The choice of the ERMs to study
- The choice of the analytes considered as stability markers for these ERMs
- The choice of acceptance criteria (AC)
- The application of statistical methods and the determination of the shelf life of ERMs, called Stability time limit (STL) for the purposes of our studies

Stability time limit (STL)

Stability is defined as a material's ability to maintain the value of its analytes at a certain level for a precise period. For users, this concept is most often close to a shelf life.

The STL, introduced as part of our stability studies on ERMs, represents the storage period during which the stability marker is considered as stable based on pre-defined regulatory and commercial aspects. The determination of STLs is estimated from specific limits, called acceptance, defined from AC.

Acceptance criteria

In order to assess stability, we apply the concept of acceptance criteria (AC), which is inspired by procedures to study the stability of medicines. The AC specifies the range within which the value of the stability marker is considered as not having changed.

This criterion is chosen empirically. In fact, it was decided arbitrarily to take a tolerance percentage around the reference value (VR).

This acceptance criterion is expressed as follow :

$$CA = \left[(1-g)\times VR\ ;(1+g)\times VR\right]$$

Where g is a coefficient whose value was determined by Bipea.

Method of modeling markers: long-term studies

Principle

This method is based on the methods generally applied for stability studies. In this paper, we will only discuss the case of linear regression. In this case we expect that the value of the analytes will not change significantly over time. The opposite case would imply that these materials are not ERMs .

Decision rules

STLs are determined using acceptance limits. The slope of the regression line is tested with a hypothesis H0 where its value varies from zero with a risk of 5% and a hypothesis H1 where it equals zero. Depending on the results from these statistical tests, two approaches can be used to evaluate the STL .

Study of the stability of samples

Approach 1. The slope equals zero with a risk of 5%.

If the slope is judged statistically equal to zero, then the RM's analyte is stable. Consequently, it cannot be assigned a shelf life. However, LINSINGER et al describe a method for estimating the stability uncertainty based on the following principle: the material changes even if it is not perceptible. Contrary to the original approach, which involved establishing a shelf life arbitrarily and calculating a corresponding stability uncertainty, for these studies the stability uncertainty has a maximum level equal to the AC used to determine the STL. In this case the STL is calculated as follows :

$$DLS = \frac{CA}{s(b)}$$

Where s(b) corresponds to the standard deviation of the regression line slope.

The calculated STL is always rounded off by default based on the corresponding unit of time. As such, the value of the stability uncertainty is also modified. Nevertheless, we believe the variance between both uncertainty value, the one equal to the AC, and the one calculated based on the determination of the chosen STL, is negligible).

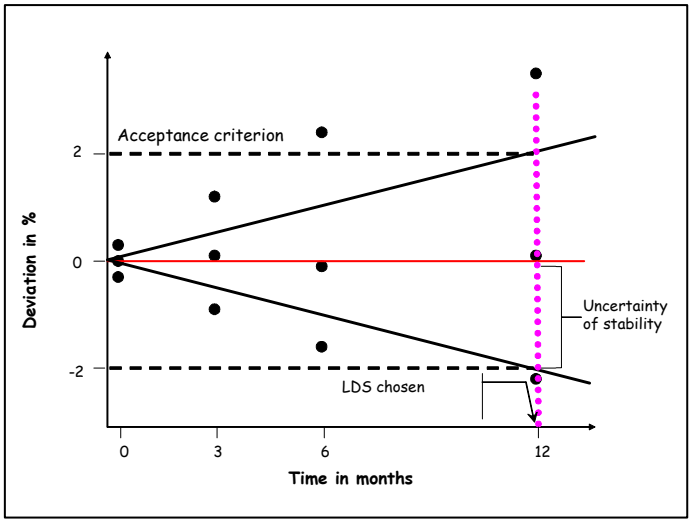


Figure 1 ♦ Schematization of a stability study using the data modeling approach.

Approach 2. The slope varies from zero with a risk of 5%.

The stability marker has changed over time. In this case, the regression model associated with a 95% confidence interval around the predicted average can be used to determine the STL. It is determined when the confidence interval cuts the acceptance limits. In all cases, the chosen STL is rounded off by default based on the unit of time used.

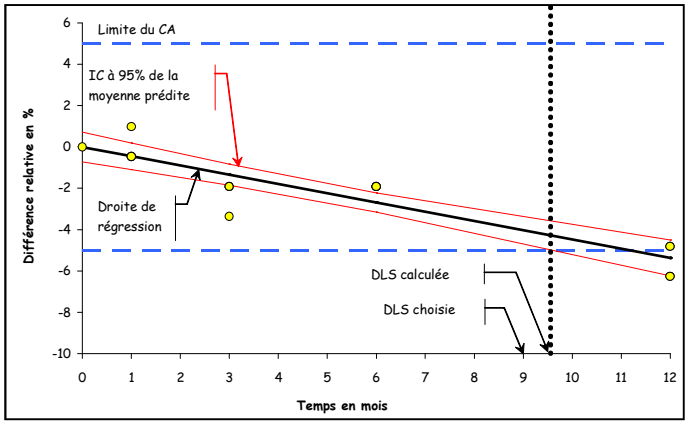


Figure 2 ♦ Example of determining the STL for the total acidity of an ERM of red wine .

Legend: Measurements are illustrated by circles (i).

[▪ ▪]: acceptance limits

Special cases:

When the STL determined by approach 1 equals twice the study time, it is then calculated with a time equal to the duration of the study plus 12 months. 12 months after the end of the study a new analysis will be performed to know whether the material can still be considered as stable. If approach 1 is used again and the STL is more than twice the total study duration, then the same approach is applied. However, the maximum stability uncertainty value still corresponds to the AC that establishes the maximum possible STL .

Julien Sarembaud
PhD student in engineering

Editor's note: *the complete bibliography for this article is available upon request from Bipea .*

LAUNCHING OF AN EXPERIMENTAL P.T. SCHEME: 51 - MICROBIOLOGY ON FOOD STUFFS

The experimental P.T. scheme will be organized based on the following principle:

- Analyses of minced meat
- Parameters analyzed:
  - Total flora
  - Total coliforms, thermotolerant coliforms, Escherichia coli and Enterobacteria
  - Salmonella spp, Clostridium perfringens, Staphylococcus aureus and Bacillus cereus
  - Listeria monocytogenes (qualitative and quantitative determination)
- 5 x 25 g. bagged samples
- sent twice during the 2007-2008 campaign (25/02/2008 and 07/04/2008) with an end of test assessment as part of a specific technical group

If you are interested in participating in this experimental campaign or would like to receive additional information, you may contact Miss Leïla Boudadi by email at: commercial @bipea.org

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Best Wishes for 2008

Bipea's various departments (1)

The Manufacturing Department

First of all, I would like to wish everyone a prosperous New Year for 2008, including many interesting projects and lots of success.

Since I am given the opportunity, I would like to introduce the Manufacturing Department, which is comprised of four technicians who are responsible for fabricating and shipping your samples so you receive them on time.

The Manufacturing Department produces 70,000 samples and ships 25,000 parcels per year while respecting our Quality procedures.

The Manufacturing Department has to continually optimize the existing manufacturing procedures by regularly purchasing material and machinery that satisfy the needs of Bipea and its members.

A new Express shipper will be put in service in 2008 along with email notification of your parcel's shipment so we can be ever more reliable in delivering your samples.

The Manufacturing Department is available to answer all of your shipment tracking questions and will contact some of you to diversify its suppliers so as not to always call on the same supplier members.

Ludovic Pirot
Manufacture Manager

The Scientific and Technical Department

The Scientific and Technical Department includes five people. It works closely with the other Bipea departments to ensure that the interlaboratory testing functions properly.

Our team participates both before and after the proficiency testing schemes, by:

- creating tests and putting forms online
- controlling the homogeneity of the stability of samples
- statistically processing data and distributing interlaboratory comparison reports
- organizing and leading commission meetings, technical groups and training courses .

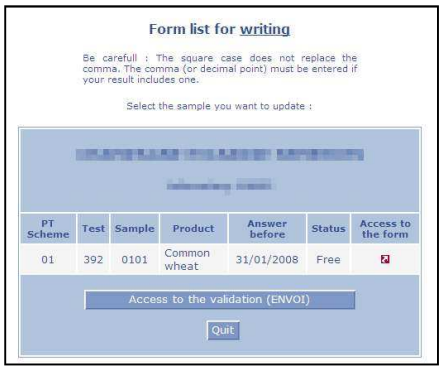
We are also available to Bipea's members to answer their questions about various issues.

Gilliane PLATERO
Scientific &
Technical Manager

Reminder about using forms

Implemented during the 2003-2004 campaign, members' response to the transmission of results by email has been very positive as shown by the results of our satisfaction survey, which shows that 80 to 90% of members are very satisfied or satisfied (depending on the question).

From this period of use, a few recurrent questions about the forms have arisen that we would like to answer through this article.



The forms' possible statuses.

The forms' status is present on the lists of forms in input and in send mode. The status can be:

- **Free:** the form hasn't been modified.
- **In Progress**: the form is partially or completely filled in. It has been saved at least once.
- **Sent:** the form has already been sent to Bipea**. This status is unique.**

Only forms with a **"Sent"** status can be integrated in the interlaboratory comparisons and participate in determining the reference value.

Correcting forms

Remember that forms can be modified up to the closing date including, of course, even forms already .

As indicated above, a form's status is unique. This means that every time a change to a "Sent" form is saved, the form's status reverts to "In Progress." **Therefore a changed form must be resent. Otherwise, the results won't be accessible to Bipea**.

Gérard Roine
IT and
Communication Manager

Since the 3rd of January 2007, **Miss Leïla Boudadi** has joined the Bipea team as a **Commercial Assistant**. Henceforth she will provide the administrative and commercial interface between Bipea and its members. We welcome here aboard.