VAMAS

www.vamas.org

VAMAS Guidelines for the Design and Operation of Interlaboratory Comparisons (ILCs)

Gert Roebben

VAMAS Report No 50
May 2017

VAMAS
www.vamas.org

Versailles Project on Advanced Materials and Standards
Enabling world trade in high technology materials since 1982

Canada | France | Germany | Italy | Japan | UK | USA | EC | Brazil | Mexico | Chinese Taipei | South Africa | Australia | Korea | India | China
1982 1983 2007 2008 2013

VAMAS Guidelines for the Design and Operation of Interlaboratory Comparisons (ILCs)

GERT ROEBBEN
European Commission
Directorate General Joint Research Centre

ISSN 1016 2186

VERSAILLES PROJECT ON ADVANCED MATERIALS AND STANDARDS

Approved on behalf of VAMAS by
Prof. Graham D Sims, (Chair)

CONTENTS

VAMAS Guidelines for the Design and Operation of Interlaboratory Comparisons (ILCs)

1 INTERLABORATORY COMPARISON (ILC)

An interlaboratory comparison is the *organization*, *performance* and *evaluation* of measurements or tests on the same or similar items by two or more laboratories *in accordance with predetermined conditions*.[1]

The three main 'variables' or 'sources of variability in the ILC results' are:

- the test method
- the laboratory (or operator)
- the test material

2 THE THREE POSSIBLE, MUTUALLY EXCLUSIVE, OBJECTIVES OF AN ILC

Every interlaboratory comparison has an aim. The ILC must be intelligently designed for reaching that aim. The aim of an ILC can be classified into one of three objectives. The three potential objectives of an ILC, which are related to the three variables mentioned above, state that an ILC may be used for assessing:

- the interlaboratory performance characteristics of new and existing methods.
- the performance of laboratories.
- one or more property values of the test material.

It is difficult and unusual to reach more than one of these objectives in a single ILC, mainly because the selection of participating laboratories depends on the ILC aim. Therefore it is recommended that only one of the general objectives listed above is addressed by the aim of the ILC. The aim must therefore be defined and clearly communicated to the potential participants prior to their enrolment in the study. By choosing the appropriate design before the comparison starts, one can avoid complicated statistical analysis with technically unjustified elimination of outlier values.

3 PARTICIPATION IN A VAMAS ILC

The general laboratory selection criteria depend on the aim of the ILC and are explained in Section 4. For VAMAS ILCs an additional requirement is to seek participation from laboratories representing all major economic areas such as represented in the VAMAS Steering Committee membership. It is requested from candidate participants to agree with the 'Participation in VAMAS Projects Terms and Conditions'.

If laboratories wish to participate in a VAMAS ILC only for their own training purposes, the chair of the relevant Technical Working Area (TWA) should judge such a wish, considering the availability of resources (e.g. test samples). A positive decision could for example be inspired by the desire to disseminate good practice to laboratories new in the field. In any case, the decision on the status of the results of this 'laboratory-in-training' (to add them to the overall data that will contribute to the method reproducibility, the assigned value of the proficiency test or the assigned value of the test material) must be made prior to the start of the measurement campaign.

4 THE BASIC REQUIREMENTS FOR THE THREE POSSIBLE OBJECTIVES OF ILC

4.1 AN ILC TO ASSESS METHOD PERFORMANCE CHARACTERISTICS

Most VAMAS ILCs aim at quantitatively assessing the reproducibility of a new method. This can be part of a larger method validation study, in which also method repeatability (within one laboratory) and method trueness (estimated as method bias against the value of a certified reference material or values obtained with another validated method) are assessed.

Statistical and other guidelines on the design of method validation studies and the analysis of their results may be found in the ISO 5725-X series of standards on 'Accuracy (trueness and precision) of measurement methods and results'.[2]

The basic requirements for the ILC design in this case are:

- To use a 'real-life' material (a material that has the main characteristics of the technically or commercially relevant range of materials that later, in daily practice, will be tested with the method).
- To use a material that is sufficiently homogeneous and stable.[3]
    - The required homogeneity level depends on the method repeatability and the expected method reproducibility: the material must be sufficiently homogeneous to be able to expose the differences between laboratories.
    - The required stability (versus temperature and time) depends on the time period foreseen between sample production and testing and on the temperatures likely to be experienced during transport and storage until future use or development as a reference material.
- To provide enough material for ≥ 2 measurements per lab; this will enable the separation of the contributions of method repeatability (within laboratories) and reproducibility (between laboratories) to the variance of the results.
- To work with qualified test laboratories (labs with demonstrated expertise in the particular field), but not only with expert laboratories, as this would lead to an overestimation of the method reproducibility.
- To agree and impose a clear and unambiguous test protocol similar to a draft standard: to assess the method reproducibility all labs have to strictly implement the method.

4.2 AN ILC TO ASSESS LABORATORY PROFICIENCY

VAMAS does not have a particular authority nor sees it as its mission to make statements about the proficiency of individual laboratories.

However, exceptionally, VAMAS may need or wish to organise a laboratory proficiency test, for the benefit of the materials testing community. This could be, for example, upon request of members which intend to meet the requirement of ISO/IEC 17025 [4] for regular participation in proficiency tests for the methods they are operating in an ISO/IEC 17025 compliant quality system. Alternatively VAMAS could allow laboratories that are not previously qualified for participation in the ILCs described under 4.1 or 4.3 to participate and thereby demonstrate their proficiency.

General and more specific statistical guidelines on the design of proficiency tests can be found in ISO/IEC 17043:2010 and in ISO 13528 'Statistical methods for use in proficiency testing by interlaboratory comparisons'.[1, 5]

The basic requirements for the ILC design in this case are, as before in 4.1:

- To use a real-life, homogeneous and stable (reference) material.
- To provide enough material for ≥ 2 measurements per lab.

Contrary to the ILC for method validation, the participation should be open to all interested laboratories, within the resource limits of the organiser. (Note: unlike VAMAS, commercial proficiency test organisers charge participants with a participation fee.)

Whether the test protocol must be flexible or strict depends on the measurand (measured property). In the case of an operationally defined measurand, the protocol must be clear and unambiguous. If the measurand is an intrinsic material property that can be assessed in multiple, different ways, then it can be left to the laboratory to choose the method.

In the evaluation of proficiency tests, it is recommended to use appropriate metrics such as:

$$z = \frac{x - X}{s}$$

z = z-score (should be < 2)
x = participant's result
X = assigned value
s = appropriate measure of variability

or

$$\zeta = \frac{x - X}{\sqrt{u_x^2 + u_X^2}}$$

ζ = zeta-score (should be < 2)
x = participant's result
X = assigned value
u_x = laboratory's estimate of standard uncertainty of x
u_x = standard uncertainty of the assigned value X

Crucial in this evaluation is the choice of the assigned value *X*, which preferably is a reference value determined by one or a number of reference labs or by using a certified reference material.

4.3 AN ILC TO CHARACTERISE A REFERENCE MATERIAL

In some circumstances within a particular VAMAS TWA an ILC is performed with the aim to determine an assigned or certified value for a reference material.

The relevant reference documents for the certification of reference materials, such as ISO 17034, are produced by ISO REMCO and ISO CASCO.[6] Basic requirements for the ILC design are:

- To use a real-life, homogeneous material, sufficiently stable for the longer term use as a reference material.
- To provide ≥ 2 samples or units per lab.
- To work with qualified test laboratories (labs with demonstrated expertise in the particular field).

Whether the test protocol can be flexible or must be strict depends on the measurand. In the case of operationally defined measurands, the protocol must be clear and unambiguous. If the measurand is an intrinsic material property that can be assessed in multiple, different ways, then it is preferable to include laboratories using different methods, as this will decrease the risk of having a method-specific bias of the assigned or certified value.

5 ADDITIONAL CONSIDERATIONS

- When working on the development and validation of a new measurement method, a route for the output into a standards development organization (e.g. ISO, IEC, CEN, ASTM…) for the final document should be identified as early as possible.
- It is helpful to prepare at an early stage a framework of the final document that the ILC is intended to produce. This framework can then be reviewed and updated as the activity progresses.
- Regardless of the aim of the ILC, it is advisable to run a smaller exercise initially with a limited number of participants. This exercise can, for example, be used to develop or select an optimised data exchange format.

- Given the nature of the VAMAS cooperation (no specific funding) it is important to keep the work expected from each participant to less than one person-week.
- Where relevant, be prepared to produce a Materials Transport Agreement (MTA), in order to clearly agree with the participants under which conditions they are provided with the test material, both from a safety as well as from an intellectual property perspective.
- If so desired, give the participants extra material to allow them to do a number of trials prior to the ultimate tests; but it must at all stages be clear whether a test is preliminary or final.
- Ensure that the instructions are written simply and clearly. When in doubt about the differences in interpretations of a certain term, one can, for example, consult the ISO Online Browsing Platform (https://www.iso.org/obp/ui/).
- Do not add too many "interesting" side issues.
- Start with no fewer than 6-8 participants to allow for a number who will not respond.
- If one laboratory submits multiple data sets (e.g. from different methods) or if results are submitted by different operators from the same laboratory or by different laboratories from the same organisation, then the independence of the corresponding data sets shall be carefully judged. If the independence is not guaranteed, then the data shall be pooled in an appropriate manner and treated as one set.
- Make it clear that you will adhere to deadlines for submission of data.
- Expect that the data analysis will probably have to cover issues not originally anticipated.
- Reports and data release should be made in a timely fashion.
- All participants should have the opportunity to review and correct a draft version of a final report.

6 ACKNOWLEDGEMENTS

The current version of these VAMAS ILC Guidelines has been produced based on previous versions to which colleagues from NPL (N. Jennett, S. Gnaniah), BAM (J. Lexow) and NIST (S. Freiman) have contributed.

7 REFERENCES

1 ISO/IEC 17043:2010 Conformity assessment - General requirements for proficiency testing (ISO/CASCO)

2 ISO 5725-2:1994/Cor 1:2002 Accuracy (trueness and precision) of measurement methods and results -- Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method (ISO/TC 69/SC 6)

3 ISO Guide 35 General and statistical principles for certification (ISO/REMCO)

4 ISO/IEC 17025:2005, ISO/IEC 17025:2005, General requirements for the competence of testing and calibration laboratories (ISO/CASCO)

5 ISO 13528 Statistical methods for use in proficiency testing by interlaboratory comparisons (ISO/TC 69)

6 ISO 17034 General requirements for the competence of reference material producers (ISO/REMCO)