Validation of test methods

General principles and concepts

Foreword

EAL and Eurolab have a Permanent Liaison Group (PLG), which is a forum where EAL and Eurolab are discussing matters of mutual interest. The PLG consists of five members from each organization.

This document has been prepared in the PLG and endorsed by both organizations.

The document is intended to give general views on certain issues related to the validation of test methods and should be seen as a common understanding and position of EAL and Eurolab. In order to define and describe the activities behind the concept “Validation of test methods” more detailed guidance documents are needed. This document should be seen as a basis for such guidance documents.

General principles to be used in validation

In the validation process an estimate is made of the representativeness, repeatability and reproducibility of the test method. The definitions are given in annex 1.

In the validation process the ultimate aim is to secure that the test methods are good enough with respect to representativeness, reproducibility and repeatability. How much effort should be spent on validation must be decided on a case by case basis. If large economic values as well as considerable health, safety and environmental issues are involved, much more emphasis must be paid to the validation of the test methods. The frequency of use of the test method should also be considered when determining the extent of validation. The total consequences of wrong results are of course larger for methods in extensive use than for test methods used occasionally.

The validation of test methods covers to a large extent the uncertainty, repeatability and reproducibility of the test method. As the factors affecting the results and contributing most to the uncertainty change from one technical sector to another or even from one test method to another, a universal solution cannot be given. Guidance on the expression of uncertainties can be found for example in the international “Guide to the expression of uncertainty in measurement” and EAL guidance document “Expression of uncertainty in quantitative testing”.

Standardized test methods should be considered validated for their intended application range and thus good enough for that purpose although their repeatability and reproducibility are not known in detail. The testing laboratory must, however, check that they apply the method correctly. For non-standardized test methods it is up to the testing laboratories to determine how far they go in defining the level of repeatability and reproducibility.

To develop a representative test method, adequate knowledge is required of the practical use of the test results and the real service conditions of the object of the test. Based on such knowledge, the “representative” properties to be determined by the test may be identified.

The factors affecting the test results and their uncertainty may be grouped into three main categories:

- Instrumental and technical factors
- Sampling
- Homogeneity
- Test method
- Equipment
Human factors

Environmental factors
- testing environment

*Instrumental and technical factors are related to the constructional and functional characteristics of the test and measurement equipment, as well as to other technical operations involved in the test (e.g., sampling, preparation of samples, test object homogeneity). Their effect may be minimized and kept under control by the following provisions:
- define the equipment as precisely as necessary
- provide a clear description of the test procedure as well as the equipment operation
- establish procedures for operational control and calibration
- ensure where applicable traceability of measurements to the SI units.

Whenever practical, the above provisions should be included in the description of the test method. References to internal procedures or applicable standards should be included.

Human factors are related to the competence of the staff and may be controlled through:
- education/basic knowledge
- on job training/practical experience

The qualification required for the personnel employed for a given test may be specified in the test method or reference can be made to the applicable internal procedures.

Environmental factors are associated to the environment where the test is performed. Among others the effect of the following parameters must be assessed and properly controlled:
- atmospheric conditions (temperature, pressure, humidity)
- pollution/contamination
- other environmental characteristics (e.g., EMC).

The effect of the above parameters should be described in the test method or reference to other applicable documents should be made. However, for new test methods this information is often not available. In some cases the data base for method validation is so large that statistical methods should be applied.

The validation process must consider the expected or required uncertainty of the test results and their intended use.

Critical threshold values (e.g., in health and environment) cannot generally be technically justified with a small uncertainty. However, if a legal limit is set, there must be test methods suited for the purpose. Reference is made to a recent ILAC Guide.

The required depth of the validation process depends also on the maturity of the test method and the prevalence of its use. One can distinguish between the following categories:
- novel methods
- methods used by several laboratories
- modification of established methods
- standardized methods

The ways, in which the validation is performed in the different cases, need not be clearly differentiated. If the fitness for purpose concept is maintained, it is often possible to validate at reasonable cost but with a higher degree of uncertainty.

The novel methods are first developed in one single laboratory, often on the basis of a special request from a customer on new ideas created in the laboratory. That customer cannot pay for a wide range validation nor can the laboratory itself. The aim of the validation of test methods must always be to demonstrate that the method is fit for the intended purpose and that the results have an acceptable uncertainty. It is important that the rules of validation of test methods do not prevent the natural technological development from taking place. The laboratory does not expect (although it does want) outside financial help for validation of novel methods and in many cases tries to protect its new development from going to its competitors or from becoming generally available to all.

When a certain number of laboratories work in the same area, cooperation and inter-laboratory comparisons can be arranged. The coordination of such activities is an extra economic burden. In order to speed up the process, external financing is needed.

The testing laboratories need to update their existing test methods. The flexible scope of accreditation as agreed between EAL and EuroLab was also intended to allow modifications to be made to accepted (accreditation covered) test methods. This requires validation procedures applicable to method modifications. It is up to the laboratories to describe their procedures for validating modified test methods.

The most thorough validation procedure is required for test method standardization purposes. The work needed is considerable and covers proficiency testing, the determination of factors affecting the uncertainty, measuring range, etc.

The financial burden cannot be laid on the laboratories but on the standardization organizations. Standardized test methods must be considered sufficiently validated for their intended application ranges. If they are not, they should be withdrawn.

The validation of test methods consists of two interrelated steps:

(i) suitability of the test to solve the problem (customer needs)
(ii) demonstration of the technical capability of the test method within the specified test range i.e. measuring the right properties with a sufficiently reliable method.

The suitability or representativeness of a test method is in many cases an attribute which is difficult to define especially for tests related to product acceptance. The test methods must be such that the results obtained correlate with the performance characteristics and operational experience of the product.

Validation procedure

Both testing laboratories and accreditation bodies are looking for procedures and guidelines for planning and controlling the test method validation process. However, the discussion above has clearly indicated that one single procedure cannot be developed. Consequently, a palette of different choices of validation techniques has to be developed. How detailed the validation will be, depends on the circumstances (needs, costs, possibilities, risks, etc.).

The validation of the test methods is, of course, of interest also to the accreditation bodies. The principle to be applied should be that the laboratory describes the way it is validating the test methods and the accreditation body should make the judgement if the procedure used is acceptable in that case. The different validation possibilities are built up around:
- utilization of calibration
- intercomparisons including the use of reference materials and reference methods
- well qualified staff and their professional judgement
- simulation and modelling
- other approaches.

Method validation is often based on the combined use of validation procedures. The validation used can be “direct” or comparative. The selection of the validation procedures should also be justified on a cost-benefit basis as long as the fitness-for-purpose is maintained. Focusing on the most critical factors affecting the test method will lead to a different solution for the validation of “exact” physical and chemical test methods as compared to that for product or subjective testing. For example, in the validation of ergonomics and sensory test methods not all possibilities are applicable.

As said above different validation procedures may be followed, their effectiveness and applicability depending on the type of test considered. They can be