## Method and Procedure Validation in Calibration Laboratories

### Background.

EUROLAB already have Cookbook no 1 which relates primarily to Method Validation in Testing Laboratories. This Cookbook focuses on Method and Procedure Validation in Calibration Laboratories. Why the difference? In many cases, Calibration Laboratories do not work to a published standard or method but are required to generate laboratory specific methods or procedures for customer equipment where there are no calibration procedures published or supplied by the equipment manufacturer. In other cases, a calibration procedure is required for a prototype product or a specialised measurement system. This means that the approach to Method and Procedure Validation is different. This document focuses on the requirements for Calibration Laboratories.

### Method vs. Procedure.

In a calibration laboratory, when we refer to a method, we typically refer to something like "The Current Volt-Drop Method" which is a generic approach to a measurement method. However, in each laboratory, there may be one or more procedures on exactly how to perform the measurement using the specific equipment of that laboratory. In other words, a procedure is a step-by-step instruction on how to execute the required task. There are however some prescribed methods that are performed by calibration laboratories especially in the Legal Metrology sector. These methods may contain step by step procedures. As these methods are generally internationally published methods, they do not generally require validation by the laboratory, especially if the laboratory has all of the prescribed equipment. On the other hand, because calibration laboratories are often required to create their own procedures, they must be validated. It should also be noted that these two terms, Method and Procedure are often interchanged, so care must be taken to understand what the laboratory is dealing with. In this Cookbook we refer specifically to laboratory generated procedures.

### Why validate laboratory procedures?

In a case where a manufacturer of equipment has provided a detailed calibration or verification procedure then it is generally not required to validate that procedure on condition the laboratory has the suitable equipment, reference standards and competent staff to do the work. This is because it is assumed that the designer of the equipment, knows best on how to perform the calibration. However, it is still good practice to perform some level of validation if only to show that the laboratory has understood the manufacturer's instructions correctly and the expected results are obtained.

Where a laboratory does not have a manufacturer's calibration procedure, then the laboratory needs to prove to itself that the laboratory generated procedure is capable of doing the assigned measurements assuring the metrological traceability within the required tolerances and measurement uncertainty. It also has to prove that the step-by-step procedure can be correctly followed by all who may use the procedure and that the same results are obtained, within reasonable variances. So even though the ISO/IEC 17025 standard clearly requires validation to be done, the laboratory would anyway need to perform the validation if they wanted to be sure that their generated procedure was "fit for purpose".

### Laboratory generated methods or procedures.

The first step is to clearly understand what is required to be done and the measurands that are involved. For example, if a calibration procedure for a specific instrument is required, then the operation, specification and acceptance limits of that instrument must be understood. Then when the laboratory is comfortable that they know how to correctly operate the instrument and understand the specifications and tolerance requirements, they need to select what reference equipment is suitable for the calibration of the instrument and the traceability it provides. A suitably qualified metrologist would then generate a step-by-

step procedure for the calibration of that instrument. After checking that the procedure is suitable, the validation phase begins.

**How to validate a laboratory procedure?**

The purpose of validation is to provide objective evidence that the Procedure can provide reliable and repeatable measurements. Consideration must also be given to it being used by different operators, different equipment and different environmental conditions. ISO/IEC 17025 requires that the laboratory should have a documented policy or procedure describing the process to be followed for the validation of their procedures. This should include the purpose, the scope, the responsibility, the data evaluation and finally the conclusion statement which must clearly state if the procedure is or is not, fit for the desired purpose.

To be sure that the acceptance criteria is achieved, a Validation Target needs to be set. This is to ensure that the resultant uncertainty of measurement is “good enough”. Ideally, a few different metrologists should follow the procedure and document the results with applicable uncertainty calculations to ensure repeatable and reproducible results. Then, using a different method (where possible), a calibration is performed, and the results are compared to that of the procedure being validated to ensure that the measurements are accurate, reliable and traceable to the SI. All of this must be documented, and reference made to the validation report in the laboratory procedure.

**When is validation required?**

Whenever the laboratory generates a new procedure or when the laboratory finds that the procedure needs to be updated because of new laboratory equipment, a change of environmental conditions or even small technical changes to the wording, then the procedure must be validated. The same applies to software modified or developed in-house that are used in the calibration process, they must be checked. This is particularly true where the laboratory has generated data capture, data reduction, use algorithms and manipulation routines including spreadsheet functions.

**Who is responsible to do the method validation?**

The laboratory that generated the procedure is ultimately responsible for the validation of that procedure as the procedure is not complete without a statement that the procedure has been validated and a reference to the validation report.

**Conclusions**

The validation of laboratory methods and procedures is often viewed as an unnecessary requirement. However, laboratories should pride themselves in producing reliable and repeatable measurements for their customers. This cannot be done without rigorous validation of laboratory methods and procedures and where applicable even standard methods.