Sample Procedure for Method Validation

1. Introduction

This is the metrology laboratory policy and procedure for developing and validating test or calibration methods when no international or national procedures are available, when deviating from standardized methods, or when no standard procedures are available.

2. Purpose

The Metrology Laboratory follows this procedure to ensure that all laboratory methods selected, modified, or developed for tests and calibrations are appropriate for the intended use, properly documented, validated, accepted by laboratory management, and agreed upon by the client. Customers of the Laboratory expect a given service to provide acceptable measurement results when they request a test or calibration. The laboratory must evaluate each method to ensure that it has qualified and competent staff, suitable facilities, equipment, and standards with acceptable metrological traceability to perform the test or calibration.

3. Responsibility

a. The Laboratory Supervisor ensures the following, in consultation with the laboratory staff as needed:
   i. Development of methods is a planned activity and assigned to qualified staff with appropriate resources.
   ii. For larger projects, plans are updated as progress is made and effectively communicated to all personnel.
   iii. Ensure the report for the test or calibration is compliant with standard requirements and customer needs.

b. The Technical Manager reviews the documented procedure, data and analysis, and recommends final acceptance to the Laboratory Manager based on the procedure assessment and analysis of measurement data.

c. The Laboratory Supervisor is responsible for final acceptance of new calibration methods.

4. Operations

a. If the laboratory does not have an appropriate method for a calibration or test, or the test or calibration requires deviation to meet the needs of the customer, the Technical Manager is notified and this procedure is implements.

b. When determining whether to proceed in developing new test or calibration method to meet the needs of a customer, the Laboratory Supervisor and Technical Manager consider at least the following factors:
   i. Availability of alternative procedures (national or international standards);
   ii. Resources of the laboratory and staffing (time, efficiency); and
   iii. Likely future demand for the service.
c. The staff conducting the Contract Review for the calibration or test must obtain a clear specification of the customer requirements and the purpose of the test or calibration including any tolerances or maximum uncertainties that are required for the artifact’s end usage (to ensure that the measurement results will be fit for purpose).

d. New methods must be developed prior to performing the tests or calibrations and contain the following information:
   i. appropriate identification (title);
   ii. scope or range of test;
   iii. description of the type of item to be tested or calibrated;
   iv. parameters or quantities and ranges to be determined;
   v. apparatus and equipment, including technical performance requirements;
   vi. reference standards and reference materials required;
   vii. environmental conditions required and any stabilization period needed;
   viii. description of the procedure, including any special items as noted in this list:
      - affixing of identification marks, handling, transporting, storing and preparation of items,
      - checks to be made before the work is started,
      - checks that the equipment is working properly and, where required, calibration and adjustment of the equipment before each use,
      - the method of recording the observations, data to be recorded, data reduction, method of analysis, and presentation of results, and
      - any safety measures to be observed;
   ix. criteria and/or requirements for approval/rejection where applicable;
   x. data to be recorded and method of analysis and presentation; and
   xi. the uncertainty or the procedure for estimating uncertainty.

5. Method Validation

a. Non-standardized methods, which include all laboratory developed methods, standardized methods modified beyond their intended scope and amplifications and modifications of standardized methods, are validated by
   i. examination to ensure completeness and compliance with requirements for essential components of metrological traceability; and
   ii. analysis of objective evidence to ensure the requirements for a specific intended purpose are fulfilled prior to use.

b. Validation methods are to be as extensive as necessary to meet the needs of their intended application. The accuracy and uncertainty of test or calibration results shall be assessed for the intended use, and shall be relevant to the clients needs.

c. Validation procedures and results are recorded, with a statement concerning the appropriateness of the new method as it pertains to the intended use.
d. Validation techniques include one or a combination of the following:
   i. Calibration/Verification using calibrated working standards;
   ii. Comparison of results achieved with other standardized methods;
   iii. Inter-laboratory comparisons when practical;
   iv. Systematic assessment of factors influencing the results; and
   v. Assessment of the uncertainty of results based on scientific understanding of the theoretical principles associated with the method and practical experience.

e. When changes are made in the validated non-standardized procedures, the influence of such changes must be documented and, if appropriate, a new validation process carried out.

f. The following types of assessments, with data and statistical analysis are examples that may be used to assess the measurement results:
   i. Inspection and technical assessment of the essential elements of metrological traceability to ensure presence and adequacy (technical review may include representatives from other laboratories, working groups, technical experts and assessors):
      i. Unbroken chain of comparisons to national and/or international standards;
      ii. Documented procedure (reviewed to ensure completeness against the list of items in Section 4.d.);
      iii. Documented measurement uncertainty (as noted in Section 4.d.v.);
      iv. Demonstrated technical competence;
      v. Reference to the international system of units (SI);
      vi. Suitable and up to date calibration intervals for standards used in the procedure; and
      vii. Adequate measurement assurance approach and supporting data.
   ii. Accuracy or Limits to Bias may use data obtained from internal testing and/or interlaboratory comparisons: t-test, normalized error (E_n), absolute or relative bias versus required tolerance limits;
   iii. Precision: standard deviation, normalized precision (P_n), F-test, comparison to required uncertainties (fit for purpose and meeting needs of the customer)
   iv. Repeatability: assessment of results over time and by different operators following the procedure as documented
   v. Reproducibility: assessment of data from other laboratories following the procedure

6. Implementation

   a. A laboratory developed test or calibration method is validated, reviewed by the Technical Manager, reviewed by the Quality Manager, and approved by the Laboratory Supervisor.
b. The method is typed and formatted into a written Standard Operating Procedure (SOP) document and assigned an identification number. The new SOP will be added to the laboratory Master List.

c. All laboratory method validation documentation is kept on file in the laboratory and maintained according to the Quality Management System.