

# GLP 1

## Good Laboratory Practice for the Quality Assurance of Laboratory Measurement Results<sup>1</sup>

Quality assurance of laboratory measurement results (measurement assurance) means understanding, modeling, measuring, and managing a measurement assurance system appropriate to the laboratory's scope of activities. Having such a system in place will allow the laboratory to know, within the limits of a measurement process, that measurement results are valid with respect to stated traceability, accuracy, and precision. A well-designed measurement assurance system provides confidence and credibility in the quality of the laboratory's measurement results by ensuring that the measurement results are metrologically traceable to appropriate reference standards and measurement units, with suitably valid uncertainties.

Quality assurance (QA) and quality control (QC) methods should consider both internal and external data for evaluating the ongoing stability and control of the measurement results and processes. At its core, the concept of measurement assurance is one of risk identification and mitigation. It provides methods for monitoring the standards and as well as the measurement process, and varying combinations of each depending on the priorities of the methods chosen.

Software quality assurance is a key function of ensuring the quality of laboratory measurement results but is outside the scope of this procedure.

### 1. Internally Obtained Measurement Assurance Data

The validity of calibrations need to be monitored with quality control procedures. Statistical techniques are used to record, analyze, and monitor charted measurement results to permit the ongoing assurance of valid and stable measurement results, integration of intermediate checks, and/or the detection of trends. The metrologists and laboratory management should also plan and review the results from quality assurance monitoring as methods are integrated into calibration procedures and during periodic management reviews and internal assessments (internal audits).

Steps taken to ensure the quality of the measurement process may include, but are not limited to:

- a. Regular use of reference materials or quality control;
- b. Regular use of alternative instrumentation that has been calibrated to provide traceable results;
- c. Functional checks of measuring and testing equipment;
- d. Use of check or working standards with control charts, where applicable;
- e. Periodic intermediate checks on measuring equipment;
- f. Replicate tests or calibrations using the same or different methods, with the use of standard deviation charts or range charts where applicable;
- g. Retesting or recalibration of retained items (e.g., customer items that are not immediately returned);

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<sup>1</sup> See also Section 7.7 of ISO/IEC 17025:2017 *General Requirements for the Competence of Testing and Calibration Laboratories*.

- h. Correlation of results for different characteristics of an item;
- i. Review of reported data by competent laboratory personnel;
- j. Intralaboratory comparisons; and
- k. Blind tests.

Each measurement parameter in the laboratory's scope of activities must be reviewed and analyzed to determine the validity of the measurement process.

The standards and the measurement process for each parameter must be in a state of statistical control. Statistical control means that the variability of the measurement process is known, stable and observed values are adequately close to reference values, within the chosen statistical limits. When a process is in statistical control and the reference values are within suitable limits, we can assume that the reported measurement uncertainties are valid.

Minimizing risks of measurement errors includes all the following laboratory functions:

- a. Training staff and evaluating effectiveness and proficiency;
- b. Monitoring the laboratory environment to minimize potential errors or excess variation;
- c. Maintaining suitable equipment (including installation, monitoring, approvals, and integrated software);
- d. Selecting and calibrating standards;
- e. Ensuring suitable suppliers for materials and calibrations;
- f. Selecting and validating procedures with evaluation of accuracy/bias and precision;
- g. Ensuring proper care and handling of laboratory standards, equipment, and items submitted for calibration;
- h. Accurately and effectively calculating, evaluating, and reporting measurement uncertainty;
- i. Participating in inter- and intra-laboratory comparisons;
- j. Creating and reviewing calibration certificates to ensure accuracy of measurement results and the effective communication of results; and
- k. Controlling data – information management (including software and information technology controls).

While other quality assurance methods could meet these objectives, the control programs developed for measurement assurance greatly increase the comprehensiveness of the program.

## **2. Externally Obtained Data Used for Measurement Assurance**

Data from measurement results obtained external to the laboratory need to be considered when evaluating the quality control and measurement assurance. Such steps that may be taken include:

- a. Evaluation of the calibration history of reference standards, working standards, and check standards;
- b. Evaluation of before/after calibrations within a laboratory to compare and evaluate results obtained from an external calibration provider;
- c. Review of historical calibration data for items calibrated having demonstrated stability;

- d. Comparison of all calibration results and calibration history on control charts for working standards and check standards with results from external calibration sources;
- e. Participation in proficiency testing using the same procedures and handling methods used for routine laboratory calibrations;
- f. Use of externally obtained data from calibrations, proficiency tests, and interlaboratory comparisons in the assessment of errors and bias in measurement results; and
- g. Participation in interlaboratory comparisons other than proficiency testing (e.g., for method validation or as a training activity).

Data from all the measurement assurance activities should be analyzed and monitored to both control and improve the laboratory's activities, if applicable. When the results of the data analyses from monitoring activities are found to be outside pre-defined criteria, appropriate actions shall be taken to prevent incorrect results from being reported to the customer.

The strength of the measurement assurance approach lies in its applicability to a wide variety of measurements with sufficient flexibility to permit each measurement control program to be tailored to the needs of a given measurement area. The sophistication of the control program depends on the criticality of the measurement.

### **3. Integration of Data from Multiple Sources**

Measurement results collected over several years may be statistically evaluated with current results being compared to results from previous years. Any observed problems or changes in the measurement results are investigated and, as appropriate, corrective action taken. Ongoing monitoring establishes a continuous and comprehensive internal laboratory measurement assurance program.

Data from internal measurement assurance programs should be compared to the results of calibration history assessments, interlaboratory comparisons or proficiency tests, and other external sources of data.

Data and analysis from the integrated assessment of measurement assurance data provides assurance of measurement quality, provides input to and validation of measurement uncertainties, supports selection and adjustment of calibration intervals for standards and laboratory instruments, and provides a graphical and statistical basis for evaluating and making decisions regarding the quality of measurement results.

### **4. NIST Office of Weights and Measures Resources**

The National Institute of Standards and Technology (NIST) provides technical guidance and support to laboratories to develop suitable measurement control programs that provide a systems approach to measurement assurance as a part of published resources and training seminars. Objectives to evaluate the entire measurement process, as a system or as a production process, will consider all the following:

- a. Personnel;
- b. Facility/accommodations;
- c. Procedures and method validation;
- d. Equipment; and
- e. Standards.

One quality tool that is often used to present the concept of monitoring the entire measurement process is a Cause and Effect Diagram where the branches represent the components on this list, examples of measurement assurance methods, and the output represents the results of a measurement process.

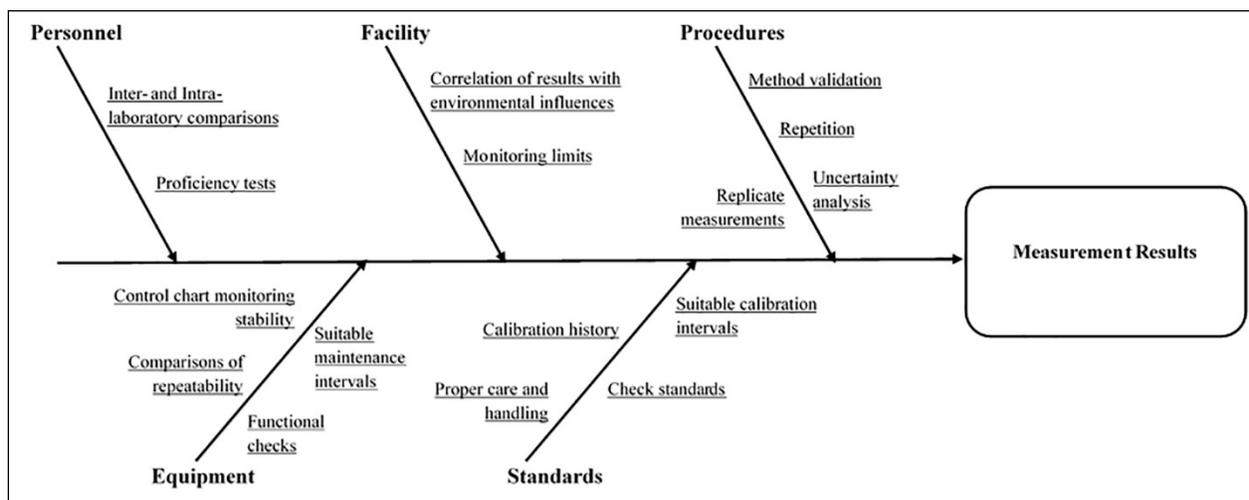


Figure 1. Cause and effect diagram with measurement assurance perspective.

Please see examples of related measurement assurance resources in Table 1.

**Table 1. Examples of Measurement Assurance Procedures and Tools Available from the NIST and the NIST Office of Weights and Measures**

Title	Description	Type of Assessments
SOP 9, Control Charts for Calibration of Mass Standards. (Includes spreadsheet job aid.)	This procedure describes procedures for the development of control charts and their use for demonstrating attainment of statistical control of a mass calibration process. The procedure may be applied to other calibration processes as well.	Procedure for: <ul style="list-style-type: none"> <li>• recommended check standards for mass calibrations;</li> <li>• establishing control charts and control chart limits;</li> <li>• monitoring and evaluating control chart data;</li> <li>• monitoring reference values;</li> <li>• transferring measurement statistics for uncertainties; and</li> <li>• checklist for evaluation of control chart design.</li> </ul>
SOP 17, Control Charts of Laboratory Owned Check Standards. (Includes spreadsheet job aids.)	This procedure may be used to develop and maintain control charts to monitor the statistical control of laboratory check standards when replicate measurements are made as a part of the standard operating procedure. This procedure may be used for volume, length, time, or other calibrations when replicate measurements are made.	Procedure for: <ul style="list-style-type: none"> <li>• establishing control charts and control chart limits;</li> <li>• monitoring and interpretation of control chart data; and</li> <li>• demonstrating example X-bar and S (standard deviation) charts and data form for replicate data.</li> </ul>
SOP 20, Standard Deviation and Range Charts. (Includes spreadsheet job aids.)	This procedure describes a process to be followed to monitor the statistical control of a measurement process using standard deviation charts or range charts for any calibration method where replicate measurements are performed and where it is not practical or feasible to maintain laboratory check standards. Standard deviation charts are preferred to the use of range charts for monitoring and evaluation process standard deviations when replicate measurements are made.	Procedure for: <ul style="list-style-type: none"> <li>• calculating initial statistics;</li> <li>• creating appropriate charts and limits;</li> <li>• using and interpreting standard deviation charts for monitoring measurement processes; and</li> <li>• demonstrating example data form for replicate evaluation of measurement results.</li> </ul>

Title	Description	Type of Assessments
SOP 30, Process Measurement Assurance Program.	The Process Measurement Assurance Program (PMAP) is used for the control and surveillance of measurement performance using check standards in measurement and calibration procedures. Incorporation of these measurement control steps ensures the validity of the measurement process and the standards used. The variables used in calculation and assignment of uncertainty can be determined and controlled using this procedure.	Procedure for: <ul style="list-style-type: none"> <li>• modeling the measurement process;</li> <li>• selecting and calibrating suitable check standards,</li> <li>• establishing initial statistics,</li> <li>• creating and preparing charts;</li> <li>• establishing statistical and reference limits;</li> <li>• evaluating process uncertainty components;</li> <li>• measuring check standards;</li> <li>• managing and evaluating the measurement process;</li> <li>• using statistical tools for ongoing evaluation; and</li> <li>• instructions on actions.</li> </ul>
All measurement SOPs for mass, volume, and length.	Calibration procedures published in NISTIR 6969 <sup>2</sup> , NISTIR 5672 <sup>3</sup> , NISTIR 7383 <sup>4</sup> , And NISTIR 8028 <sup>5</sup> .	All Standard Operating Procedures (SOP) incorporate a section on measurement assurance methods specific to that calibration procedure.
Measurement Assurance System Evaluation Form.	Used for laboratory recognition and/or accreditation to evaluate measurement assurance programs. Should be integrated as a part of the laboratory Quality Management System.	Provides an outline for systematic evaluation of a laboratory measurement parameter, including a series of questions to document a periodic assessment.
PT Follow-Up Form.	Originally developed as a form associated with NISTIR 7214. Used for annual assessment of laboratory PT results. Should be integrated as a part of the laboratory Quality Management System.	Provides a framework for a systematic review of proficiency testing results.

<sup>2</sup> NISTIR 6969, *Selected Laboratory and Measurement Practices, and Procedures to Support Basic Mass Calibrations*, Georgia L. Harris.

<sup>3</sup> NISTIR 5672, *Advanced Mass Calibrations and Measurements Assurance Program for the State Calibration Laboratories*, Kenneth L. Fraley, Georgia L. Harris.

<sup>4</sup> NISTIR 7383, *Selected Procedures for Volumetric Calibrations*, Georgia L. Harris.

<sup>5</sup> NISTIR 8028, *Selected Laboratory and Measurement Practices and Procedures for Length Calibrations*, Jose A. Torres, Georgia L. Harris.

<b>Title</b>	<b>Description</b>	<b>Type of Assessments</b>
PT Reporting 4-year Analysis Form.	Used for laboratory recognition and/or accreditation assessments and tracking. Should be integrated as a part of the laboratory Quality Management System.	Form for tracking laboratory results on a rolling 4-year basis with plans for the coming year. Includes space for tracking corrective actions, preventive actions, and improvement actions.
Method Validation Administrative Procedure (Sample).	This sample metrology laboratory policy and procedure is used 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. Should be integrated as a part of the laboratory Quality Management System.	Outlines responsibility and authority for method validations and approvals; includes: <ul style="list-style-type: none"> <li>• procedures for method validation;</li> <li>• types of assessments that should be utilized to assure the quality of measurement results; and</li> <li>• a sample evaluation form that can be used to document the assessment of new/laboratory procedures;</li> <li>• use of replicate tests or calibrations using the same or different methods;</li> <li>• use and evaluation of check standards; and</li> <li>• use of interlaboratory comparisons.</li> </ul>

Title	Description	Type of Assessments
<p>NIST IR 6969<sup>6</sup>, Section 8, Statistical Techniques. Associated with Section 9, Reference Tables.</p>	<p>Includes statistics that are used in metrology to summarize experimental data, to provide the basis for assessing its quality, and to provide a basis for making probabilistic decisions in its use.</p>	<p>Includes:</p> <ul style="list-style-type: none"> <li>• calculation of standard deviations;</li> <li>• pooling standard deviations,</li> <li>• estimates of standard deviation from range data;</li> <li>• estimates of within and between standard deviation;</li> <li>• determining confidence and statistical tolerance intervals;</li> <li>• using F-test, t-test, and Welch Satterthwaite equation for estimating effective degrees of freedom; and</li> <li>• using random numbers.</li> </ul>
<p>NIST/SEMATECH e-Handbook of Statistical Methods<sup>7</sup></p>	<p>Web-based reference that helps scientists and engineers incorporate statistical methods into their work as efficiently as possible. Serves as a reference on experimental design and appropriate data analyses when a statistician is not available to help. Serves as a useful educational tool to help users of statistical methods and consumers of statistical information better understand statistical procedures and their underlying assumptions, and more clearly interpret scientific and engineering results stated in statistical terms.</p>	<p>Includes 8 Chapters:</p> <ol style="list-style-type: none"> <li>1. Exploratory Data Analysis.</li> <li>2. Measurement Process Characterization.</li> <li>3. Production Process Characterization.</li> <li>4. Process Modeling.</li> <li>5. Process Improvement.</li> <li>6. Process or Product Monitoring and Control.</li> <li>7. Product and Process Comparisons.</li> <li>8. Assessing Product Reliability.</li> </ol>

<sup>6</sup> NISTIR 6969, *Selected Laboratory and Measurement Practices, and Procedures to Support Basic Mass Calibrations*, Georgia L. Harris. Note: originally part of NIST Handbook 145, *Handbook for the Quality Assurance of Metrological Measurements*, John K. Taylor and Henry V. Oppermann, 1986, which is out of print).

<sup>7</sup> NIST/SEMATECH *e-Handbook of Statistical Methods*, <http://www.itl.nist.gov/div898/handbook/>, January 19, 2018.