GMP 11

Good Measurement Practice for Assignment and Adjustment of Calibration Intervals for Laboratory Standards

1. Introduction

1.1. Purpose

Measurement processes are dynamic systems and often deteriorate with time or use. The design of a calibration system would be incomplete without some established means of determining how often to calibrate instruments and standards. A calibration performed only once establishes a one-time reference of uncertainty. Recalibration detects uncertainty growth and serves to reset values while keeping a bound on the limits of errors. A properly selected interval assures that an item will be recalibrated at the proper time. **Proper calibration intervals allow specified confidence intervals to be selected and they support evidence of metrological traceability.** The following practice establishes calibration intervals for standards and instrumentation used in measurement processes.

Note: GMP 11 provides the basis for documenting calibration intervals. This GMP is a template that must be modified beyond Section 4.1 to match the scope[1] and specific measurement parameters and applications in each laboratory. Legal requirements for calibration intervals may be used to supplement this procedure, but are generally established as a maximum limit assuming no evidence of problematic data.

1.2. Prerequisites

1.2.1. Calibration history and valid calibration certificates for all laboratory standards.

1.2.2. Up-to-date measurement assurance and uncertainty data for all measurement processes in the laboratory that match the laboratory Scope.

1.2.3. Expected tolerance limits where applicable.

1.3. Safety

1.3.1. No outstanding safety concerns

1 The laboratory scope includes the measurement parameters, range of nominal values, associated uncertainties, and calibration and/or test methods.

2. Methodology

2.1. Summary

Recommended calibration intervals are based on various examples of traceability as described in GMP 13. As data is collected and evaluated, the laboratory technical manager may adjust the calibration intervals to ensure that measurement results are not invalidated by the intervals selected according to this procedure.

2.2. Apparatus

None.

2.3. Procedure

2.3.1. Identification of Parameters

The laboratory must identify all measurement parameters associated with the Scope of calibrations and tests that will be performed in the laboratory. All standards used in a calibration process must be assessed to determine their level of significance.

2.3.2. Standards are identified as being critical parameters or secondary parameters.

2.3.2.1. Critical Parameters

Components that contribute more than 25 % of a measurement's uncertainty are identified as critical parameters. To ensure an accurate evaluation of performance, calibration intervals are determined to meet a 99 % reliability target.

2.3.2.2. Secondary Parameters

Components that contribute less than 25 % but more than 1 % of a measurement's uncertainty are identified as secondary parameters. Secondary parameters are assigned calibration intervals designed to meet a 95 % reliability target.

2.3.3. Assignment of Initial Intervals

Assignment of initial intervals is based on examples and recommendations given in this GMP. Otherwise, an initial interval is equivalent to the amount of time that passes before a metrologist makes the first observation of a result lying outside the warning limits of a control chart when the investigation yields no apparent correctable cause or when results from

proficiency tests fail the normalized error assessment (or when the observed bias exceeds the laboratory uncertainty), or the maximum authorized through law or technical review.

2.3.4. Initial Intervals – Additional Standards

A calibration interval must be documented for all standards used in the laboratory to comply with the definition and interpretation of metrological traceability. Statements such as "as needed" are not acceptable alone without additional qualifications. A statement such as "calibrations are conducted on an annual basis and may be extended based on demonstrated control through use of control charts, surveillance testing, or interlaboratory comparisons, but will not exceed five years" would be acceptable.

2.3.5. Absence of Control Charts or Measurement Assurance Data

If no initial measurement assurance data is available, the laboratory's Technical Manager may assign initial intervals based on evidence observed in the calibration certificate history, engineering evidence, manufacturer′s specifications, NIST recommendations, and valid technical experience.

2.4.Adjustment of Intervals

2.4.1. Intervals will be adjusted when determined to be necessary by the laboratory's Technical Manager. Subsequent intervals may be adjusted based on analysis of measurement assurance data, demonstrated control through surveillance assessments, and ongoing stability as demonstrated through interlaboratory comparisons or proficiency tests.

2.4.2. The intervals may be adjusted by performing a technical analysis, taking the following factors into consideration as appropriate, and documenting the assessment:

- calibration history;
- measurement assurance data;
- data for the population of similar standards, equipment or technologies;
- NIST recommendations;
- statistical analysis methods; and
- manufacturer′s recommendations.

2.4.3. Inadequate reasons for adjusting intervals might include failure to have an adequate system for tracking due dates, lack of funding for suitable calibrations, or loss of competent staff. Extending calibration intervals must be based on valid, technically supported, and documented assessments.

3. Assignment of Uncertainty

The uncertainty associated with the establishment of calibration intervals is not included as a part of the uncertainties associated with the respective measurement SOPs. See SOP 29 for calculating uncertainties for standards and measuring instruments.

4. Calibration Intervals for the Laboratory Scope

4.1. As this document is a template, the laboratory may eliminate all examples in this section and reference another laboratory document that combines metrological traceability information and calibrations in one file as long as it completely states calibration intervals. It is NOT recommended to include calibration intervals in the laboratory Quality Manual so that updates can regularly be implemented when calibrations are performed without excessive document approval requirements.

4.2. Example for Mass

4.2.1. Mass Critical Parameters

4.2.1.1. Balance and Measurement Process Performance

Balance service, maintenance, and calibration must be a regular part of the laboratory operations and is generally performed on a regular periodic basis (often annually) unless problems are observed.

Balance performance in weighing processes following SOP 5 and SOP 28 (e.g., Echelon I) is evaluated in each measurement series. An F-test ratio evaluates the observed standard deviation of the process against the accepted standard deviation of the process.

All other weighing processes have incorporated measurement control procedures and control charts that must be evaluated for balance performance characteristics as data is collected.

Table 1. Balances used for mass (and gravimetric volume) calibrations.

Item	Initial Service and Calibration Interval (months)	Source
Balances and Mass Comparators	12	Approved Provider

4.2.1.2. Mass Standards

Each weighing series in Echelon I also incorporates a check standard with a value that is evaluated using a t-test. The observed value is compared to the accepted reference value to determine if there might be problems. Mass

standards are dynamic with use. Wear, contamination and other factors can cause drift from accepted values. Thus, the following intervals have been set:

Table 2. Example mass standard calibration intervals only where Echelon I is in place.

Item	Initial Cal Interval (months)	Source
R 1. kg and R 1.. kg	48	NIST
S_c 1. kg and S_c 1.. kg	(alternating 2 years) 48	NIST
R 100. g and R 100.. g	48	NIST
S_c 100. g and S_c 100. g	(alternating 2 years) 48	NIST
R 30 kg to R 2 kg	12	Lab
R 500 g to R1 mg	12	Lab
S_c 500 g to to S_c 1 mg	12	Lab
W 25 kg to W 1 mg	12	Lab
R 500 lb to 1 µlb	12	Lab
W 5000 lb to 1 µlb	12	Lab
R = Reference; S_c = check/control; W = working standards		

Note: where Echelon I is not in place, all reference standards or working standards may need to be calibrated by a suitable calibration provider.

4.2.2. Mass Secondary Parameters

Availability of multiple units for environmental equipment enable the laboratory to conduct internal comparisons immediately after a calibration or between calibration cycles and generate internal calibration reports to enable extension of calibration intervals as long as adequate stability is demonstrated. See procedure for adjusting calibration intervals. The following table contains initial calibration intervals.

Table 3. Example calibration intervals for environmental equipment.

Item	Initial Cal Interval (months)	Source
Barometer	12	Accredited Lab
Hygrometer	24	Accredited Lab
Thermometer (digital)	12	Accredited Lab

4.3.Example for Length

4.3.1. Length Critical Parameters

4.3.1.1.Length Standards

Length standards are dynamic with use. Wear, contamination and other factors can cause drift from accepted values. The following intervals have been set due to these factors:

Table 4. Calibration intervals for length standards.

Item	Initial Cal Interval (months)	Source
100 ft Tape #1	60	NIST
100 ft Tape #2	60	NIST
25 ft or 7 m Tape	60	NIST
18 in Steel Rule	120	NIST
Length Bench	24 (if used or moved)	Lab

4.3.2. Length Secondary Parameters

Table 5. Calibration intervals for length secondary parameters.

Item	Initial Cal Interval (months)	Source
Thermometer (digital)	12	Accredited Lab

4.4.Example for Volume

4.4.1. *Volume Critical Parameters*

4.4.1.1.Volume Standards

Volume standards are dynamic with use. Wear, contamination and other factors can cause drift from accepted values. Seals and valves must regularly be assessed for leaks and stability. Initial calibration intervals are as follows:

Table 6. Calibration intervals for volume standards.

Item	Initial Cal Interval (months)	Source
R 100 gal standard	60	NIST
R 25 gal standard *	60	Lab
R 5 gal standard *	12	Lab
Glassware- Autopipetes 5 L to 100 mL	120	Lab
*Gravimetric calibration for volumes 5 gallon or smaller, and all "slicker plate" standards. Laboratory must be qualified for performing gravimetric calibrations. Volume transfer is acceptable above 5 gallon. *May be a "slicker plate" type. None are hand-held, "dump" style, test measures.		

4.4.2. Volume Secondary Parameters

Table 7. Calibration intervals for volume secondary parameters (gravimetric).

Item	Initial Calibration Interval (months)	Source
Barometer	12	Accredited Lab
Hygrometer	24	Accredited Lab
Thermometer (digital)	12	Accredited Lab

Table 8. Calibration intervals for volume secondary parameters (transfer).

Item	Initial Cal Interval (months)	Source
Thermometer (digital)	12	Accredited Lab

4.5.Example for Thermometry

4.5.1. *Thermometry Critical Parameters*

4.5.1.1.Temperature Standards

Temperature standards are dynamic with use. Shock, contamination and other factors can cause drift from accepted values. Recalibration intervals are as follows:

Table 9. Calibration intervals for temperature standards.

Item	Initial Cal Interval (months)	Source
25.5 ohm SPRT	36	NIST
100 ohm PRT′s	12	Accredited Lab
Standard Thermistor	12	Accredited Lab
Check Standards	12	Accredited Lab
Liquid-in-glass standards*	6*	Accredited Lab

**Annual inspection must also ensure that there is no damage or separation in the liquid column. See NIST SP-1088 (2009)2 for additional maintenance plan requirements. New thermometers should be checked at least once a month at the ice point for a minimum of the first six months of use.*

Where internal intrinsic or reference standards are available, and the laboratory has the demonstrated competency, the following may be used to set or adjust calibration intervals: triple point cells, melting point cells, and ice baths (using documented procedures).

2 NIST SP 1088, Maintenance and Validation of Liquid-in-Glass Thermometers, Christina D. Cross, Dean C. Ripple, W. W. Miller, Gregory F. Strouse, January 01, 2009, http://www.nist.gov/manuscript-publication-search.cfm?pub_id=900914.