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 receive recalibration at the proper time. **Proper calibration intervals allow specified confidence intervals to be selected and they support measurement traceability.** The following practice establishes calibration intervals for standards and instrumentation used in measurement processes.

1.2. Prerequisites

1.2.1. Calibration history for laboratory standards

1.2.2. Expected tolerance limits if applicable

1.3. Safety

1.3.1. No outstanding safety concerns

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.

2.2. Apparatus

None.

2.3. Procedure

2.3.1. Identification of Parameters

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. Critical parameters are checked and defined below:

Mass Critical Parameters (The example provided below corresponds to Option A in Mass Traceability in GMP 13.)

Balance Performance

Balance performance in Echelon 1 weighing processes is evaluated in every measurement series. An F-test ratio evaluates the observed standard deviation of the process against the accepted standard deviation of the process. The balance performance component is also tested with a check standard in each weighing series. The check standard value is evaluated with a t-test by compared the observed value to the accepted reference value. All other weighing processes have incorporated measurement control procedures and control charts that are evaluated as data is collected.

Mass Standards

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 1. Calibration intervals for mass standards

Item	Initial Cal Interval (months)	Source
P1. kg + P1.. kg	48	NIST
C1. kg + C1.. kg	(alternating 2 years) 48	NIST
P30 kg - P2 kg	12	Lab
P500 g - P1 mg	6	Lab
C500 g - C1 mg	6	Lab
W25 kg - W1 mg	12	Lab
P Pound Standards	24	Lab
W Pound Standards	12	Lab
P = primary; C = check/control; W = working standards		

Length Critical Parameters

Length Comparison Performance

The measurement performance of each length calibration is evaluated with a check standard (when performing tape-to-tape comparison).

Initial Length Intervals

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 2. 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

Volume Critical Parameters (Example shown corresponds to Option A for volume calibration in GMP 13.)

Volume Comparison Performance

The measurement performance of a volume transfer calibration is evaluated in each use with a repeatability check. Use of check standards is the preferred method for evaluating the measurement process over time. Traceability for volume standards may be established through gravimetric calibrations using traceable mass standards.

Initial Volume Intervals

Volume standards are dynamic with use. Wear, contamination and other factors can cause drift from accepted values. Calibration intervals are as follows:

Table 3. Calibration intervals for volume standards

Item	Initial Cal Interval (months)	Source
P100 gal standard	60	NIST
P25 gal standard *	60	Lab
P5 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.

Temperature Critical Parameters

Temperature Comparison Performance

The measurement performance of each temperature comparison calibration is evaluated with a check standard and can be verified periodically using triple point cells, melting point cells, and ice baths (using documented procedures).

Initial Intervals

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

Table 4. Calibration intervals for temperature standards

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

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. Secondary parameters are defined below:

Mass and Gravimetric Volume Secondary Parameters

Environmental Measurement Equipment

Table 5. Calibration intervals for environmental equipment

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

Length Secondary Parameters - No secondary parameters.

Volume Secondary Parameters

Water Temperature Measuring Device

Table 6. Calibration intervals for volume secondary parameters

Item	Initial Cal Interval (months)	Source
Thermometer	12	Lab

Temperature Secondary Parameters - No secondary parameters.

3. Calculations

3.1. Initial Intervals

3.1.1. Assignment of Initial Intervals

Assignment of initial intervals is based on these recommendations. Subsequent intervals may be adjusted based on analysis of Check standard data that is recorded on control charts. The 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 the chart when the investigation yields no apparent correctable cause. Warning limits are established at ± 2 standard deviations of the measurement process around the accepted value of the check standard.

3.1.2. Absence of Control Charts

If no control charts are available, the laboratory's Technical Manager will assign the initial interval based on engineering evidence, manufacturer's specifications, NIST recommendations, and experience.

3.2. Adjustment of Intervals

3.2.1. Intervals will be adjusted when determined to be necessary by the laboratory's Technical Manager.

3.2.2. The intervals will be adjusted by taking the following factors into consideration as appropriate:

- calibration history;
- measurement assurance data;
- data for the population of similar equipment;
- NIST recommendations;
- statistical analysis methods; and
- manufacturer's recommendations.

4. 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 the instruments.