SOP 17

Standard Operating Procedure for Control Charts of Laboratory Owned Check Standards

1 Introduction

1.1 Purpose

This procedure may be used to develop and maintain control charts to monitor the statistical control laboratory owned check standards.

1.2 Prerequisites

1.2.1 The procedure to be monitored must match the calibration procedure that is used.

1.2.2 Either a check standard at each nominal value is used or a set of check standards are selected to monitor the range of items that are calibrated by the laboratory.

2 Summary

A check standard is obtained and calibrated several times initially to establish a reliable mean value and to estimate the standard deviation of calibration. All such calibrations are made using the applicable procedure that is used for calibration. A reference value may be obtained using a better calibration than the one that will be monitored. Directions for preparing and using a control chart for monitoring the mean ($\bar{x}$) and the standard deviation and range chart are given. The $\bar{x}$ control chart monitors the process with respect to both the standard and the variability, while the standard deviation or range chart monitors its short-term precision. When the calibration process is determined to be in a state of statistical control, the calibrations made at that time may be considered to be valid and the process standard deviation may be used, as appropriate, to calculate the uncertainty for the calibrations using SOP 29.

Note: If a full evaluation of the process bias is desired, it is best if the reference value of the check standard are provided by an outside laboratory accredited to perform the applicable calibrations.

3 Equipment

3.1 A check standard is required, and should be constructed of similar materials and design as the standards under calibration.

3.2 All equipment designated in the applicable SOP.

4 Procedure

4.1 Initial Measurements

4.1.1 Calibrate the check standard a minimum of 12 times to establish the baseline chart. A calibration is defined as the result of duplicate measurements as required by the SOP (i.e., a complete test consists of two runs). Calibrations may be made on successive days, but no two complete tests should be made on any single day. Note: 25 to 30 points are recommended to determine uncertainties.

4.1.2 Tabulate the measurement data using the notation and a form such as the one contained in the Appendix of this SOP. The data may be maintained in a spreadsheet or other electronic program in lieu of a paper form.

4.1.3 A standard deviation may be calculated for each set of runs according to the appropriate SOP with a pooled standard deviation determined for the measurement process. This is preferred.

4.1.4 Calculate the mean of the two trials $\bar{x}_i$ and the ranges between runs. The ranges R_i, are the absolute differences between run 1 and run 2 for the n tests. Be sure that only absolute values are used in the determination of the range and average range!

4.1.5 Calculate the average range $\left|\overline{R}\right|$ of the trials, for the n tests as follows:

$$\overline{R} = \frac{\sum |R_i|}{n} \qquad \text{Eqn. 1}$$

Estimate the standard deviation of the process s_p, for each set of made runs according to the SOP.

4.1.6 The standard deviation may be calculated using the average range as follows (obtain values for d_2^* from NISTIR 6969 Table 9.10 and see NISTIR 6969 Section 8.3 for additional notes):

$$s_p = \frac{\overline{R}}{d_2^*} \qquad \text{Eqn. 2}$$

4.2 Construction of Control Charts (See also SOP 9)

4.2.1 Construct the following control charts using the data of section 4.1.

4.2.2 Construct an $\bar{x}$ control chart for a check standard with the following control limits:

Reference value (when available)

Central Line = $\bar{\bar{x}}$ (mean of the average values)

Lower warning limit (LWL) = $\bar{\bar{x}} - 2s_p$

Lower control (or action) limit (LCL) = $\bar{\bar{x}} - 3s_p$

Upper warning limit (UWL) = $\bar{\bar{x}} + 2s_p$

Upper control (or action) limit (UCL) = $\bar{\bar{x}} + 3s_p$

4.2.3 Construct a Standard Deviation or Range chart using the same approach. However, you may use 2 and 3 as the respective multipliers for the Upper Warning Limit and Upper Control Limits. Note that there will be no negative numbers when calculating standard deviations.

4.2.4 Construct an *R* (range chart) control chart for duplicate measurements having the following control (or action) limits. Note that *R* (the range) and |*d*| (absolute difference of duplicate measurements) are equivalent for duplicate measurements.

Central Line = $\bar{R}$ (average range)
LCL = LWL = 0
(There should be no negative numbers recorded when using absolute values!)
UWL = 2.512 $\bar{R}$
UCL = 3.267 $\bar{R}$

4.2.5 These limits are *t* values for 95 % and 99.7 % confidence intervals for a sample size of 30.

4.2.6 The recommended format for construction of *R* control charts is given in NISTIR 6969, Section 7.4.

4.3 Use of Control Charts

4.3.1 An appropriate check standard is calibrated each time the laboratory performs calibrations using the SOP. If the calibrations extend over several days, the check standard is calibrated daily. The values of $\bar{x}$ and s_p or *R* for each calibration of the check standard are plotted on the respective control charts, preferably in sequential order. The limits on the charts are such that 95 % of the values should fall within the warning limits and rarely should a value fall outside of the control limits, provided that the system is in a state of statistical control.

4.3.2 If the plotted value of $\bar{x}$ lies outside of the control limits and the corresponding value on the standard deviation or range chart is within the control limits, a source of systematic error is suspected.

4.3.3 If the values for the standard deviation or range chart fall outside of the warning limits but inside of the control limits, a decrease in precision is indicated. Other problems should be investigated.

4.3.4 No calibration data should be accepted when the system is out of control.

4.3.5 If the plotted values for either $\bar{x}$, s_p or R are outside of the warning limits but inside of the control limits, a second set of duplicate calibrations should be made. If the new values are within the warning limits, the process may be considered to be in control. If they lie outside of the warning limits, lack of control is indicated. Corrective actions should be taken and attainment of control demonstrated before calibration measurements are considered to be acceptable.

4.3.6 Even while the system is in an apparent state of control, incipient troubles may be indicated when the control data show short- or long-term trends, shifts, or runs. The *t*-test and *F*-test may be used to assess the significance of such observations (see NIST IR 6969 Section 8.9, 8.10, and 8.11).

4.4 Interpretation of Control Chart Data

4.4.1 Demonstration of "in control" status indicates that the calibration process is consistent with the past experience of the laboratory. That is to say, there is no reason to believe that excessive systematic error or changes in precision have occurred.

4.4.2 To the extent appropriate, the precision of measurement of the check standard may be extended to the calibration of other standards of similar nominal size made by the same measurement method.

4.4.3 Extension of the s_p for the check standard to other calibrations assumes that all aspects of its calibration correspond to those for the other calibration.

Appendix
Control Chart Check Standard Data

Check Std ID _____ Nominal Value _____

Test Number	Date	*Run 1*	*Run 2*	*Average of Runs*	Range* $\lvert d\rvert$ = |Run 1 - Run 2| (Max – Min)	Standard deviation**
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
SUM						
				$\sum \bar{x}$	$\sum \lvert d\rvert$	pooled std dev:

n*** = _____

$\bar{R} = \frac{\sum |R|}{n}$ = _____ UWL = 2.512 $\bar{R}$ =

UCL = 3.267 $\bar{R}$ =

* This is the range, *R*, of the two trials and is actually the larger value minus the smaller value.
** Use of the standard deviation and pooled standard deviations are preferred to the use of range as an estimate of the standard deviation.
****n* is the number of tests used to calculate the control limits.