

SOP 20
Standard Operating Procedure
for
Standard Deviation and Range Charts

1 Introduction

1.1 Purpose

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 evaluating process standard deviations when replicate measurements are made.

1.2 Prerequisites

The procedure must match the calibration procedure that is being monitored.

2 Summary

Because of the size and cost of some laboratory standards, it is not always practical to have check standards remain in the laboratory for the purposes of measurement control. However, it is practical to maintain standard deviation (s_p) charts or range (R) charts for each nominal size standard that is submitted to evaluate the standard deviation of replicate runs. Directions for preparing and using an R control chart that monitors the precision of the test procedure are given. It is assumed that standards of the same nominal capacity and design will have similar characteristics with respect to the repeatability of tests. Since it is not practical to run a sufficient number of tests on each unknown standard to determine the repeatability, the absolute difference between replicate tests are graphed on the same s_p or R chart to reflect the repeatability of measurement of the unknown standards tested in the laboratory. Note: Provers of similar readability may be grouped together on the same chart as long as results from different sized provers pass an F-test (e.g., provers greater than and equal to 500 gal generally have 20 in³ graduations and equivalent repeatability).

3 Equipment

All equipment is designated in the respective calibration SOP.

4 Procedure

4.1 Define: Determine appropriate measurement assurance

The SOP used for calibration should contain instructions to define the type of measurement assurance and check standards to be measured and analyzed to ensure the process and standards are in control prior to reporting measurement values from calibrations.

4.2 Measure: Data Collection

4.2.1 Conduct the required number of replicates per test on the unknown test item per the designated SOP. A minimum of 12 sets of data must be available before a reasonably adequate data base is established. Note: 25 to 30 replicate sets of data are needed to determine valid uncertainties.

4.2.2 Tabulate the measured errors as determined by each of the two trials using a form such as the one contained in the Appendix. It is recommended that the data be maintained in a spreadsheet or other electronic program in lieu of a paper form. (If the unknown item is adjusted after the first run, the first trial reading after the adjustment is used in the evaluation).

4.3 Analyze: Calculate the standard deviation (recommended)

4.3.1 A standard deviation of the replicate runs, s_p , may be calculated for each set of runs according to the appropriate SOP. An accepted standard deviation over time is calculated as the pooled standard deviation to represent the accepted standard deviation of the measurement process. This pooled standard deviation may be used to perform F-tests as part of the SOP to immediately determine pass/fail status of the runs.

4.4 Analyze: Calculate the range (optional)

4.4.1 Where the range is used, it represents is the absolute difference of two (or more) replicate runs. Be sure that only absolute values are used in the determination of the range and average range!

$$R = |x_1 - x_2| \quad \text{Eqn. (1)}$$

4.4.2 Calculate the average range of the trials, \bar{R} , for the n tests as follows:

$$\bar{R} = \frac{\sum |R|}{n} \quad \text{Eqn. (2)}$$

4.4.3 The estimated standard deviation may be calculated using the average range as follows (obtain values for d_2^* from NISTIR 6969, Table 9.1 and see NISTIR 6969, Section 8.3 for additional notes). For one set of two runs, the d_2^* value is 1.414; for one set of three runs the d_2^* value is 1.91:

$$s_p = \frac{\bar{R}}{d_2^*} \quad \text{Eqn. (3)}$$

4.5 Analyze: Construct Appropriate Charts

4.5.1 Construct Standard Deviation Charts and Range Charts using the same approach. You may use 2 and 3 as the respective multipliers for the Upper Warning Limit and Upper Control Limits on standard deviation charts for replicates of two. See Table 1 for limits and replicate options based on a Warning Limit of 95 % and an Action Limit of 99.73 %. Note that there will be no negative numbers when calculating standard deviations or ranges. The formula for calculating alternative limits for standard deviations is given by:

$$UCL = s_{pooled} \sqrt{F_{\alpha, J-1, K(J-1)}} \quad \text{Eqn. (4)}$$

Where the alpha is chosen based on the desired limits (e.g., 0.05 for 95 %); $J-1$ represents the degrees of freedom from the replicates used in the applicable SOP; and $K(J-1)$ represents the degrees of freedom corresponding to the pooled standard deviation.

Table 1. Warning and Control Limits for Standard Deviation Charts.

Replicates	Upper Warning Limit	Upper Action (Control) Limit
2	1.960	3.00
3	1.731	2.432
4	1.614	2.172
5	1.540	2.016
10	1.371	1.675

4.5.2 Construct an R control chart having the following limits based on the number of replicate measurements used in the process where the value in the table is multiplied by the average Range, \bar{R} or Standard Deviation:

Table 2. Warning and Control Limit Multipliers for Range Charts.

Replicates	Upper Warning Limit	Upper Action (Control) Limit
2	2.512	3.267
3	2.050	2.575
4	1.855	2.282
5	1.743	2.115
10		1.777

The lower control limit is zero. Note: Using subgroup standard deviations and pooled standard deviations is preferable.

4.6 Interpret the Data: Using Control Charts

4.6.1 Replicate trials are run on each standard submitted to the laboratory for calibration according to the specific SOP. The values for standard deviation or range are plotted on the appropriate control chart, preferably in

chronological order. The limits of the charts are such that 95 % of the values should fall within the warning limits and rarely should a value fall outside the control limits, provided that the system is in a state of statistical control.

- 4.6.2 If the values plotted on the standard deviation or range chart fall outside of the control limits, a decrease in precision is indicated. Problems with the standards or process will need to be investigated.
- 4.6.3 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.

4.7 Controlling the Process

- 4.7.1 No calibration data should be accepted when the system is out of control.
- 4.7.2 If a plotted value for standard deviation or range is outside of the warning limit but inside the control limit, a second set of replicate calibrations should be made. If the new value for the standard deviation or range is within the warning limit, the process may be considered in control. If it lies outside of the warning limit, lack of control is indicated. Investigation should be made, with suitable corrective action taken with demonstrated attainment of control before calibration measurements are considered acceptable.
- 4.7.3 Demonstration of "in control" indicates that the calibration process is consistent with the past experience of the laboratory. There is no reason to believe that excessive changes in precision have occurred.
- 4.7.4 The accuracy is inferred from a consideration of control of the sources of bias.
- 4.7.5 To the extent appropriate, the indicated measurement precision of the calibration of the measured type of standards may be extended to the calibration of other standards of similar type, capacity and design.

Appendix

Standard Deviation and Range Data

Standard Capacity: _____

Laboratory: _____

Test Number	Date	<i>Run 1</i>	<i>Run 2</i>	<i>Average of Runs</i>	Standard deviation ^A	Range ^B Run 1 - Run 2 (Max - Min)
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
^A Use of the standard deviation and pooled standard deviations are preferred to the use of Range as an estimate of the standard deviation.						
^B This is the range, <i>R</i> , of the two trials and is calculated as the larger value minus the smaller value.						

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