SOP 30

Recommended Standard Operating Procedure for a Process Measurement Assurance Program

1. Introduction

1.1. Purpose

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 by the use of this Standard Operating Procedure (SOP). See also NISTIR 6969, SOP 9, and NISTIR 7383, SOP 17 and 20.

1.2. Prerequisites

1.2.1. A Standard Operating Procedure that describes and provides rigor and consistency in the calibration process.

1.2.2. Check standards that represent the standard and/or the items to be calibrated. Check standards must be stable and their values should be established with accuracy, since they will be used to control the uncertainty in the calibration process. Check standard reference values must be determined by a higher level of calibration than the procedure for which the standard will be used and preferably by an independent process or laboratory competent to work at that level. As a minimum, the check standard may be calibrated within the laboratory if they are qualified to work at the next higher level and use a procedure one level higher than the calibration process to be controlled.

1.2.3. The operator must be experienced in the calibration process and the standard operating procedure for the calibration to which this method is applied. The operator should also have had specific training on SOP 29 (uncertainty identification and calculations).

1.2.4. A calculating system for statistical control that calculates standard deviations, control limits, F-tests, t-tests, root-sum-of-the-squares (RSS), and creates control charts.

2. Methodology

2.1. Scope, Precision, Accuracy

This method can be used on any measurement or calibration process where a check standard can be substituted for, or measured as, the item being calibrated. The method duplicates the standard operating procedure with the check standard serving as a surrogate for the item being calibrated. The results of these check standard measurements are recorded, charted, and analyzed to establish the measurement capability and to set process control and warning limits. The limits are used to establish process uncertainties and to control future measurement performance.

2.2. Summary

Process Measurement Assurance Program (PMAP) is used for determining and controlling the measurement process uncertainty as the calibration is performed. The uncertainty includes effects of the measurement instrument, the operator, the procedure, the standards, and the environment. Each application is modeled to meet the following objective for determining and controlling uncertainties 1) in the measurement process; 2) in the calibration standards; or 3) in both the process and the standards.

The check standard is selected to evaluate the standard deviation of the process, s_p, other process uncertainties, u_o such as material density or air density accuracy, and possible bias, u_d of the process (see SOP 29). Reference measurements for the check standard are performed after calibrating the reference or working standards and after the servicing of the measurement instrument.

Control measurements of the check standard are graphed for visual examination of process performance and are evaluated against control reference limits.

Statistical tools, F-tests, and t-tests, determine if there are process changes in variability and bias (systematic error). These tests are used when process performance is questionable; when current data is evaluated to establish new reference control limits; and when evaluating uncertainty statements.

2.3. Apparatus/Equipment Required

2.3.1. A calibration process that meets the requirements of the standard operating procedure (SOP) is required.

2.3.2. A check standard must be selected to evaluate the established objectives of monitoring: the 1) measurement process uncertainty; 2) the calibration standards; or 3) both the process and the standards.

2.3.3. A data input, statistical calculating, and control charting system to provide analysis of measurement control data (i.e., special software package or spreadsheet).

Table 1. Symbols description

Symbol	Description
S_c	Control measurement of check standard
S_{cs}	Accepted value of check standard
U	Expanded Uncertainty (of the process)
u_c	Combined standard uncertainty
u_s	Standard uncertainty of the standard
u_o	Standard uncertainty of other factors
u_d	Standard uncertainty of differences
s_p	Standard deviation of the process
k	Coverage factor

2.4. Procedure

2.4.1. Preliminary Procedure

2.4.1.1. Model the Calibration Process

Set objectives for the PMAP application. These objectives will establish the value of the check standard, the check standard measurement procedure, and influence the frequency of control measurements of the check standard. Objectives may be: 1) Determine the standard deviation of the process (s_p); 2) Determine the Expanded Uncertainty (U); 3) Measure the value of the calibration standard uncertainty (u_s). The model may allow any one objective or a combination of the objectives to be established with a single PMAP application and PMAP control chart.

Diagram the process to clarify the measurement steps and determine the approach that will achieve the established objectives. When determining and controlling the expanded uncertainty (U), evaluate the range of use of the process to ensure that the check standard values will determine the variability and the maximum bias that result from the process and the calibration standard. Determining and controlling expanded uncertainty requires duplicating the calibration process and determining where in the process to insert the control measurement using the check standard. When the objective of PMAP is to control the uncertainty of the calibration standard (u_s), the PMAP measurement may vary from the calibration process to allow inserting the check standard measurement close to the measurement of the calibration standard. Diagram the calibration process to establish how many check standards and PMAP applications are required to meet objectives. Also, diagram the process to determine where in the process to insert the control measurements of the check standard.

2.4.1.2. Select and Calibrate Check Standard

For calibration process uncertainty determination and control, select a check standard that approximates the item to be calibrated. The selected check standard should be selected to evaluate maximum random variation, $s_{p,}$ and bias of the process. The check standard selected should also be used to evaluate other objectives, u_o, of the specific calibration process. For multiple ranges of use, a check standard and PMAP application will be required for various portions of the range. For example, Double Substitution (SOP 4) will require, at a minimum, a check standard for each decade (1000 g, 100 g, 10 g, 1 g, 100 mg, and 10 mg) on each balance used. The selected check standard, S_c, should be calibrated to establish its accepted value with an uncertainty level sufficient to control the calibration process uncertainty. The calibration of the check standard must be completed using a standard which is independent of the calibration standard that the PMAP process is designed to control.

For control of the calibration standard, use a check standard that is not part of routine measurement and that will evaluate the changes in the calibration standard's accepted value, not the maximum random variation of the process. The check standard used to control the calibration standard should be used less frequently (less than ¼ as often) than the calibration standard. For example, in (SOP 28), using Design A.1.2 (a 4-1 weighing design) at 1000 g, requires the selection of a 1000 g check standard that is measured less frequently than the item under test in order to evaluate the two 1000 g calibration standards. The selected check standard should be calibrated using a calibration standard other than the calibration standard(s) it will be controlling.

2.4.2. Establish Initial Reference Limits

2.4.2.1. Control measurements to establish initial reference limits may be made at any time to verify current measurement process performance. But to achieve control of calibration standards accuracy and measurement instrument capability, the control measurements should be made just after calibration of the calibration standards and servicing of the measurement instrument. Any significant change in the calibration status can then be detected by the performance change in the reference limits data.

2.4.2.2. Make the control measurement by duplicating the calibration process with the check standard substituted for the calibrated item. Make an observation of the check standard and determine its calculated value by completing calculations as described in the calibration SOP.

2.4.2.3. Record the calculated value of the check standard and plot it on the control chart and evaluate it with reference to the accepted value of the check standard. Record the date, time, and information tags with data.

2.4.2.4. Evaluate the bias (difference) between the mean of the measured check standard values and the accepted value for S_c from its calibration report. A t-test may be used to assess individual values (observed versus accepted).

Bias (deviation) of check standard = Observed mean of S_c - Accepted S_c

2.4.2.5. Repeat the control measurement at various intervals to sample environmental change and other factors than can affect measurement performance. Although a control chart and some statistical control can be established with as few as seven to 12 measurements, a minimum of twenty-five is recommended for estimates and control of uncertainties.

2.4.3. Create and Prepare Control Charts

2.4.3.1. Construct a graph with the deviation of the check standard measurements on the y-axis and chronological date and time (or observation number) on the x-axis. The accepted value of the check standard is identified near the center of the chart.

The y-axis of the control chart should extend plus and minus three standard deviations from the mean $(\bar{x})$ of the control measurements. Control measurements may be charted as deviation from the accepted value of the check standard or as the observed measurement result, or as a calculated correction. However, care must be taken to understand the impact of updating calibration values for the standards on the data maintained in the charts. (For example, it is generally preferred to plot the calculated mass or conventional mass rather than to track the history of the observed differences to another standard or instrument.)

2.4.3.2. Establish control chart parameters by calculating the mean and the estimate of the standard deviation, s_p of the check standard reference measurements. Control chart parameters to establish limits are as follows:

$$\text{Reference Line} = \text{Accepted } S_c$$

$$\text{Mean Line} = \bar{x} \text{ of Observed } S_c \text{ data}$$

$$3s \text{ Action Limits} = \bar{x} + 3\,(s_p)$$

$$2s \text{ Warning Limits} = \bar{x} + 2\,(s_p)$$

$$\text{Process Bias} = \text{Mean of Observed } S_c - \text{Accepted } S_c$$

2.4.4. Establish Reference Limits and Process Uncertainty

2.4.4.1. Establish reference control limits (as described in 2.4.3.2) by calculating control limits and process bias using the control measurements obtained when calibration standards and measurement instruments are calibrated. These limits are to be stored and used as a reference for future control measurements. Future control measurements should be control-charted and tested against these limits for “in” or “out” of control status. This reference data will also be statistically used to periodically evaluate process and calibration standard performance for change from the calibrated reference status.

2.4.4.2. Establish the expanded uncertainty (U) by using the reference data, standard uncertainty of the standard, u_s, and any other standard uncertainty, u_o not covered by the reference data according to the calibration SOP.

$$u_c = \sqrt{(s_p)^2 + (u_s)^2 + (u_o)^2 + (u_d)^2}$$

$$U = k * u_c$$

According to the PMAP model, s_p, u_o, and u_d are evaluated by comparison with the reference data. The uncertainty for the standard, u_s, and, in some situations, additional u_o are included in PMAP calculation of the process expanded uncertainty (U).

2.4.5. Measure Check Standards

2.4.5.1. Control measurements of the check standard should be made periodically to ensure that the current measurement performance remains in control of the established reference limits. Control measurements should be tested for "in" or "out" of control status and charted on the control chart. The frequency of the control measurements is dependent on the objective of the application.

When the objective is to determine and control the calibration process uncertainties, control measurements should be made during the calibration process to ensure the calibration results are accurate and within reference uncertainty statements. Control measurements should be made prior to returning calibrated items to the customer. A minimum of 25 control measurements are required within the calibration period or interval assigned to the calibration standards and the calibration process.

When the objective is to control calibration standards, working standards, or primary standards, the frequency of control measurements should be less than ¼ the use of the standard being controlled. The reduction in measurements ensures that the check standard receives less use and wear than the standard being controlled. Control measurements are charted and the Process Bias (observed mean value – accepted) is evaluated to detect any significant change in the calibration standard being controlled.

2.4.6 Manage and Evaluate Process Performance at Specific Intervals

2.4.6.1. Examine each control measurement data point as it is charted. Evaluate each data point for its control status and investigate causes for out of control data. Analyze the measurement process uncertainty, including the process standard deviation, s_p, process bias, and other uncertainties quarterly or every five to ten data points to ensure that significant changes in uncertainties do not occur.

2.4.6.2. Evaluate current performance using the control measurements described in 2.4.5 to establish the current standard deviation of the process, s_p, process bias and other uncertainties. Perform each evaluation at specific calibration intervals established by the calibration of standards and service of the measurement instrument. This evaluation, referred to as "calibration" of the process, is performed at intervals that will ensure detection of changes in the calibration uncertainty statements.

2.4.6.3. Use statistical tools at specific calibration intervals to evaluate current data performance to reference data that was established at calibration of standards and at the service of the measurement instrument. This evaluation will assist in deciding when to recalibrate calibration standards and service the measurement instrument.

Use the F-test to evaluate if a significant change in the standard deviation or process performance has occurred.

$$F - test = \frac{(s_{p\ \text{large}})^2}{(s_{p\ \text{small}})^2}$$

The current and previously established reference standard deviations are compared and evaluated using F-test table values based on degrees of freedom in the measurements.

Use the t-test to evaluate if a significant change in measurement process bias has occurred.

$$t - test = \left| \frac{(Bias_{\text{new}}) - (Bias_{\text{old}})}{\sqrt{\frac{(s_{p\ \text{new}})^2}{n_{\text{new}}} + \frac{(s_{p\ \text{old}})^2}{\text{n}_{\text{old}}}}} \right|$$

The current (new) and previous (old) reference bias is compared and evaluated using t-test table values based on the degrees of freedom in the measurements. Keep track of the signs and trends associated with bias over time.

2.4.6.4 Take action based on the results of statistical evaluation. If F-tests and t-tests reveal no significant change in process performance, use the current data analysis to establish new process reference limits, control chart and uncertainty statements (as described in 2.4.3 and 2.4.4). If the process has a stable history, it is permissible to pool the current data with previous reference limits to establish new reference limits, control chart, and uncertainty statements. Continue control measurements until the next calibration interval.

If F-tests and t-tests reveal significant change in the measurement process, investigate the specific cause. If the cause for the change cannot be identified and corrected, then collect new reference data to establish new reference limits and process uncertainties. If a specific cause is found and corrected, and subsequent control measurements indicate an "in-control" status, continue collecting control measurements and test against established reference limits until the next calibration interval.

2.4.7. Continue the measurement assurance procedure as described in 2.4.2 through 2.4.6 to determine and control the measurement process capability.

Significant changes in the measurement process capability can result from the following:

- measurement procedure change;
- measurement instrument change;
- calibration standards change; and/or
- location change.

These changes can require repeating the procedure (from 2.4.2 through 2.4.6).