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# KAN GUIDE ON MEASUREMENT ASSURANCE

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## 1. INTRODUCTION

Chapter 5.9 of SNI 19-17025-2000 "General Requirements for the competence of testing and calibration laboratories" on "*Assuring the Quality of Test and Calibration Results*" requires:

> *" The laboratory shall have quality control procedures for monitoring the validity of test and calibration undertaken, the resulting data shall be recorded in such a way that trends are detectable and, where practicable, statistical techniques shall be applied to the reviewing of the results. This monitoring shall be planned and reviewed and may include, but not limited to:*
>
> *a. regular use of certified reference materials and/or internal quality control using secondary reference material;*
> *b. participation in inter-laboratory comparison or proficiency testing programs;*
> *c. replicate tests or calibration using the same or different methods;*
> *d. retesting or recalibrating of retained items;*
> *e. correlation of results for different characteristics of an item*
>
> *note: the selected method should be appropriate for type and volume of the work undertaken "*

This document gives the recommended method to implement the above requirement that is applicable for calibration and testing laboratories that willing to be accredited by National Accreditation Body of Indonesia (KAN) based on SNI-19-17025-2000.

## 2. MEASUREMENT ASSURANCE CONCEPT

Measurement assurance is a process to ensure adequate measurement results that may include, but is not limited to:

- the use of good experimental design principle so that the entire measurement process, its components and relevant influence factors can be well characterized, monitored and controlled;
- complete experimental characterization of the measurement process uncertainty including statistical variations, contribution from all known, or suspected influence factors, imported uncertainties and the propagation of uncertainties throughout the measurement process;
- continuously monitoring the performance and state of statistical control of measurement process with proven statistical process control techniques, including the measurement of well characterized *check standard* along with the normal workload and use of appropriate *control chart*

The purpose of statistical control is to guarantee the 'goodness' of measurement results within predictable limits and to validate the statement of uncertainty of the measurement result. Statistical control methods can be used to test the measurement process for change with respect to bias and variability from historical levels. However, if the measurement process is improperly specified or calibrated, then the control procedures can only guarantee comparability among measurements.

The assumptions that relate to measurement process apply statistical control; namely that the errors of measurement are uncorrelated over time and come from a population with a single distribution. The tests for control depend on the assumption that the underlying distribution is normal (Gaussian), but the test procedures are robust to slight departures from normality. Practically speaking, all that is required is that the distribution of measurements be bell-shaped and symmetric.

Measurements on a *check standard* provide the mechanism for controlling the measurement process. Measurement on a check standard should produce identical results except for the effect of random errors, and tests for control are basically tests of whether or not the random errors from the process continue to be drawn from the same statistical distributions as the historical data on the check standard. Changes that can be monitored and tested with the check standard database are:

- changes in bias and long-term variability; and
- changes in instrument precision or short-term variability

The concept of statistical process control is based on comparing how measurement process is performing today (often a single measurement) to how it has performed in the best (a database of measurement results). From our knowledge of statistics, we have an idea what to expect from a stable process.

In a stable measurement process, we expect the majority of measurement to fall within control limits that were established based on statistical evaluation of historical data. Measurements that are fall outside the control limits are assumed to be “out of control”. An investigation is then needed to find the cause(s) and suitable corrective action must be taken.

Bias and long-term variability are controlled by monitoring measurements on a check standard over time. A change in the measurement on the check standard that persists at a constant level over several measurement sequences indicates possible:

- change or damage to the reference standards;
- change or damage to the check standard artifact;
- procedural change that vitiates the assumption of the measurement process

A change in the variability of the measurement on the check standard can be due to one of many causes such as:

- loss of environmental controls;
- change in handling techniques
- severe degradation in instrumentation

Short-term variability or instrument precision is controlled by monitoring standard deviations from repeated measurements on the instrument(s) of interest. The database can come from measurements on a single artifact or representative set of artifacts.

The artifacts must be of the same type and geometry as items that are measured in the workload, such as:

- items from the workload
- a single check standard chosen for this purpose
- a collection of artifacts set aside for this specific purpose

Calibration and testing can be thought as a production process in which the measurement result and resulting report is the final product, then *PLAN – DO – CHECK – ACT (PDCA)* process flow, which was originally developed for production process and is often used to monitor change and to measure improvement of process may be applied in assuring calibration and testing results.

The implementation of PDCA cycle in a measurement assurance system may be described as follows:

- PLAN: identify the measurement process and procedures; use modeling tools to help diagram the process; select and calibrate appropriate check standard
- DO: collect initial data to characterize the process; make sure that data are good, stable, accurately reflect reference values and under control, use checklist and data collection form to ensure consistency in gathering data; plot initial data on a control chart; and establish limits

- CHECK: periodically gather additional data and immediately plot them on control chart; monitor data on ongoing basis
- ACT: evaluate and analyze the data; use statistical tools for consistency; implement corrective action or improvement as needed or desired; at this point, the cycle continues.

EXAMPLE:

*PLAN:*

*a. Define the measurement process: We are establishing a measurement assurance program for the calibration of 200 g OIML $F_1$ class. We use a double substitution procedure using a 1 kg capacity AT 1005 comparator with 0.01 mg readability and will use a 1 kg OIML $E_2$ class reference standard for the calibration*

*b. Establish the objectives: remember that whenever possible it is a good idea to monitor both variability of the measurement process and the value of the reference standard*

*c. Diagram (model) the process: the model of calibration process can be described as follows:*

| | |
|---|---|
| *S* | *: reference standard* |
| *X* | *: unit under test* |
| *X* | *: unit under test* |
| *S* | *: reference standard* |

*The duplication of the above calibration process for a check standard can be described as:*

| | |
|---|---|
| *S* | *: reference standard* |
| *Sc* | *: check standard* |
| *Sc* | *: check standard* |
| *S* | *: reference standard* |

*We can use one check standard to monitor this process because we can't incorporate check standard into the measurement and achieve the goals of the procedure, we will duplicate the process by substituting a check standard into the place of unit under test and repeating the process for a check standard.*

## 3. THE USE OF CHECK STANDARD

Check standard is a standard that is used as part of measurement assurance program to provide check on the standard and process to ensure that the standards, measurement results, and measurement processes are within acceptable statistical limits.

The check standard should be thought of in terms of a database of measurements. It can be defined as an artifact or as a characteristic of the measurement process whose value can be replicated from measurements taken over the life of the process. Examples of check standard are:

- measurements on a stable artifact;
- differences between values of two reference standards as estimated form calibration experiment;

- values of a process characteristics such as bias term, which is estimated from measurement on reference standards and/or test items.

To control the measurement process and determine a valid uncertainty, we should select a check standard that:

- duplicate the item being tested/calibrated;
- evaluates the maximum random variation and bias of the process over an extended period of time;
- has an established mean or calibrated reference value, with and uncertainty statement; and
- its calibrated values is independent from the reference standards to ensure that bias due to the process or calibration standards may be detected.

EXAMPLE:

*PLAN:*

*d. Select and calibrate check standard: For the measurement described before in the example, we will choose a standard that will monitor bias, and determining the variability of the process. We have an older set of stainless steel standards that have demonstrated stability over time, and have an independent calibration value from the other accredited calibration laboratory. In this case we use a 200 g stainless steel mass standard of* $F_1$ *class from this set to be a check standard for the calibration described in this example. The calibration value of this standard is used as the reference value for this check standard.*

## 4. DEFINING AND CREATING CONTROL CHART

Control chart is a graphical tool used to visualize data when monitoring, evaluating and improving a measurement process:

- monitoring and evaluating may include the need for controlling process through corrective action and process improvement
- the measurement process includes the evaluation of many contributing factors including standards, measurement, variability, uncertainties, environmental condition and staff performance

The type of chart, which is commonly used, is called a *Shewhart Control Chart* or *a Variable Control Chart* that is fairly intuitive and easy to implement. It is characterized by the plotted measurement values, X (variables), a centre line, e.g. mean or reference value based on calibration, and upper/lower warning/control limits. This type of chart is good for detecting large changes but not as good for quickly detecting small changes (of the order of ½ to 1 standard deviation) in the process.

From the definition, a control chart is used for monitoring, evaluating and improving the measurement process, using control chart allow us to:

- ensure agreement with reference value, or to detect bias, offset, or change in the set point of references;
- ensure ongoing stability of process variability and reported uncertainty or detect change in the variability of the process that affects the uncertainty analysis;
- ensure and document ongoing stability of the standards and process for accuracy and traceability and to predict future measurement values;
- make decision regarding corrective action or process improvement

The question is often asked in defining and creating control chart, "how many initial measurements do we need? ", considering our knowledge about statistic and the measurement process, we can answer the question as follows:

- 2 to 3 measurements, we need at least 2 measurement to calculate mean and standard deviation, however, only this amount of data provides limited values for our process
- 7 to 12 measurements, we need at least 7 to 12 data to obtain initial estimates on sample and population to begin developing control chart. The first set of 7 to 12 measurements will be used to establish initial reference limits. 10 is good number of data points to use as the baseline for our initial control measurements. Be sure to gather data over a period of time, with not more than 1 measurement from the same day
- 25 t0 30 measurements, we need at least 25 to 30 data points on which to have statistical decision about the data. We will need this many points to establish whether the data are stable, randomly distributed and symmetric about the mean. We should also have at least this much data for estimating and calculating measurement uncertainty
- 50 to 100 measurements, once we have maintained data for a while, it is good idea to keep at least 50 to 100 data points or at least 3 years data to allow monitoring trends. We will need to determine and document how long data will need in our laboratory and the basis for archiving our old data

There is specific information that should always be on a control chart and some information should be always obtained with the data and maintained in a database. The data collection forms and a spreadsheet template or specialty software program should be used to gather complete information consistently. There are some essential information and good information to have in a control chart:

- <u>Essential information</u>:
  1. *Titles*: identify laboratory, standard operating procedure, standard(s) and/or check standard(s), nominal value, and time of measurement
  2. *Data*: measured or calculated values, number of data point(s), mean, standard deviation
  3. *X-axis*: identify observations by date or time
  4. *Y-axis*: observations or calculated values, with measurement unit identified
  5. *Central line*: mean, and reference value whenever available, though it may not be at the center
  6. *Limit*: identification of upper and lower warning and control (action) limits.

- <u>Good information:</u>
  1. *Legend*: when more than one series present
  2. *Tolerances*: when applicable
  3. *Uncertainties*: for reference value, check standard and process output
  4. *Equipment information*: device readability, configuration setting
  5. *Standard information*: calibration date and interval information
  6. *Responsible staff*: need on chart or in database
  7. *State of Control*
  8. *Information about previous limits and history of the chart/data*: if available

It is critical to understand the concept associated with each following step in creating control chart no matter what tools are used to create the chart. Various tools may be used, but the most common are spreadsheet or a specialty software product created for this application. The following is the explanation to create a control chart, with an example on the measurement assurance of the mass standard calibration using Microsoft Excel Spreadsheet:

EXAMPLE:

*DO:*

*a. STEP 1: Make measurement*

*We will collect observations and perform calculations and will start to create control chart when we have 10 corrections (mass values) to enter. Remember that we need 7 to 12 data points to construct the initial chart. We do not plot balance observations, but the correction (mass value) of the check standard because we want to evaluate the entire measurement process and not just the balance variation over time.*

*We should normally enter data in columns when using a Microsoft Excel Spreadsheet as follows*

| | |
|---|---|
| description | : 10 points - control chart initial data |
| SOP | : direct comparison |
| balance used | : AT 1005 / 0,01 mg |
| load | : 200 g |
| unit | : mg |
| check standard | : stainless steel F1 OIML class |
| measurement process | : calibration of 200 g F1 OIML class |

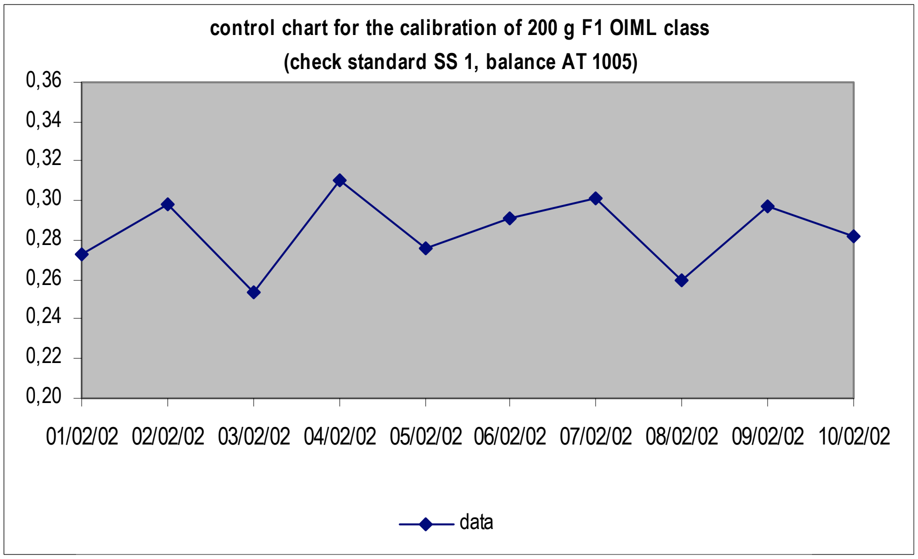
| date | correction | reference |
|---|---|---|
| 01/02/02 | 0,27 | 0,276 |
| 02/02/02 | 0,30 | 0,276 |
| 03/02/02 | 0,25 | 0,276 |
| 04/02/02 | 0,31 | 0,276 |
| 05/02/02 | 0,28 | 0,276 |
| 06/02/02 | 0,29 | 0,276 |
| 07/02/02 | 0,30 | 0,276 |
| 08/02/02 | 0,26 | 0,276 |
| 09/02/02 | 0,30 | 0,276 |
| 10/02/02 | 0,28 | 0,276 |

*b. STEP 2: Plot values*

*Plotting values on the y – axis versus dates of measurements on the x – axis is the standard approach for plotting chart values*

*Plotting values in a spreadsheet will require (at a minimum) selecting column measurement data for series 1 and the chart type and then select the dates of measurement for the x – axis.*

*This is the simplest approach to creating a chart. We will need to enter labels and titles next. Be sure to label your values with the appropriate measurement units. To create control chart in Microsoft Excel, you should use "LINE chart".*




*c. STEP 3: Calculate statistics*

*The initial statistics in creating a control chart are central tendency and dispersion.*

*The mean and standard deviation are used to represent the central tendency and dispersion. Spreadsheet allows us to calculate these statistics more easily than using calculator. However, it will help to be able to validate our spreadsheet equation with a calculator.*

| | |
|---|---|
| description | : 10 points - control chart initial data |
| SOP | : direct comparison |
| balance used | : AT 1005 / 0,01 mg |
| load | : 200 g |
| unit | : mg |
| check standard | : stainless steel F1 OIML class |
| measurement process | : calibration of 200 g F1 OIML class |

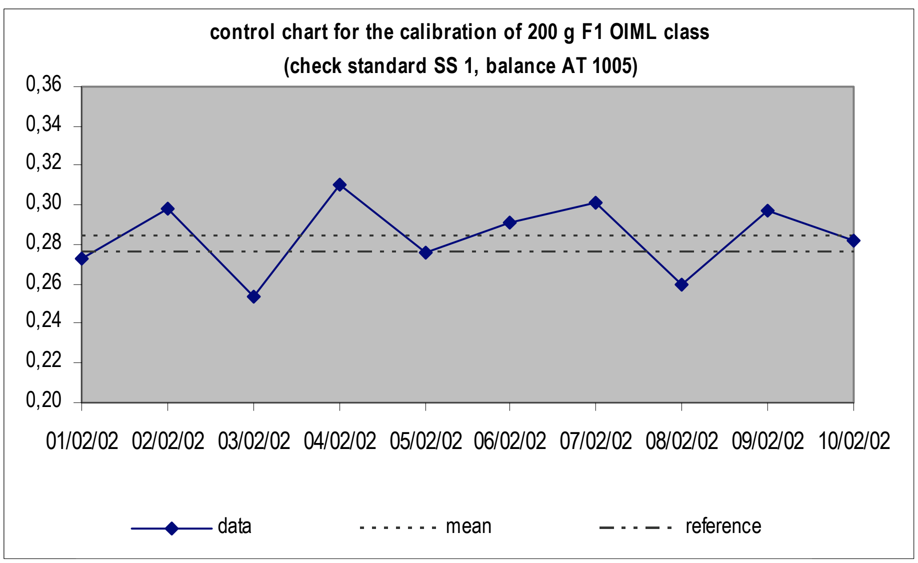
| date | correction | reference | mean | std dev |
|---|---|---|---|---|
| 01/02/02 | 0,27 | 0,276 | 0,28 | 0,02 |
| 02/02/02 | 0,30 | 0,276 | 0,28 | 0,02 |
| 03/02/02 | 0,25 | 0,276 | 0,28 | 0,02 |
| 04/02/02 | 0,31 | 0,276 | 0,28 | 0,02 |
| 05/02/02 | 0,28 | 0,276 | 0,28 | 0,02 |
| 06/02/02 | 0,29 | 0,276 | 0,28 | 0,02 |
| 07/02/02 | 0,30 | 0,276 | 0,28 | 0,02 |
| 08/02/02 | 0,26 | 0,276 | 0,28 | 0,02 |
| 09/02/02 | 0,30 | 0,276 | 0,28 | 0,02 |
| 10/02/02 | 0,28 | 0,276 | 0,28 | 0,02 |

*d. STEP 4: Plot "mean" and "reference value"*

*The centre line for a control chart is often used as, or represents, an accepted value. As a minimum, we plot the mean value of the data to show the central tendency. But, also having an independently calibrated value allows us to evaluate the reference standard and process for the presence of bias.*

*The accepted value may be the mean values as determined from the data, may be calibration value determined by another laboratory, or may be a calibration value determined in-house using a higher level procedure than the one being monitored. If the reference value is the mean of corrections, we can only monitor variability in the process and change of the standard, no monitoring of bias is possible.*

*If the reference value comes from a higher-level independent laboratory, bias from reference value can be monitored as well as process variability. Having a reference value with the fewest measurement possible in traceability chain (e.g. from an NMI) will provide a measure of bias with the highest level of confidence. If the reference value comes from a higher level measurement in-house, bias within the laboratory can be monitored also as well as process variability, but with lower level of confidence that is possible when using an independence reference value from an NMI or higher level accredited calibration laboratory.*




*e. STEP 5: Establish and plot limits*

*In a control chart, we may use statistical limits and/or specification limits. Once limits have been calculated, they are plotted on the chart. In spreadsheet, we will enter the value for the limit in a column and enter a new series on the chart with the values from the column.*

*Statistical limits are based on probability distributions we calculate and use warning limits and action limits to determine if the process is in a state of statistical control (i.e. producing consistent, stable output). Action limits are also called control limit, we use to indicate that some type of action is required when data is outside the control limits.*

*Statistical warning limits, are calculated as:*

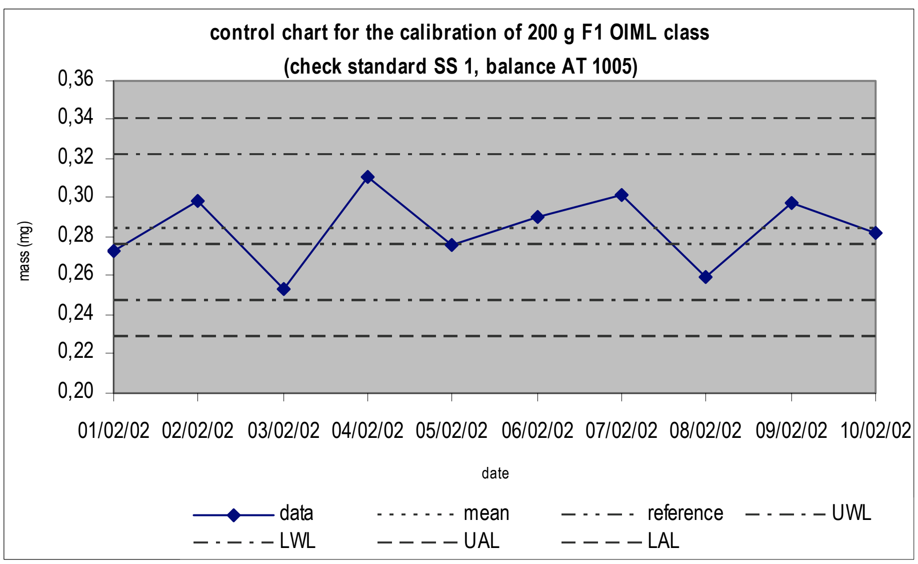
*Upper Warning Limit (UWL) = mean + (2 x standard deviation)*
*Lower Warning Limit (LWL) = mean – (2 x standard deviation)*

*Statistical action (control) limits, are calculated as:*

*Upper Action Limit (UAL) = mean + (3 x standard deviation)*
*Lower Action Limit (LAL) = mean – (3 x standard deviation)*

description : 10 points - control chart initial data
SOP : direct comparison
balance used : AT 1005 / 0,01 mg
load : 200 g
unit : mg
check standard : stainless steel F1 OIML class
measurement process : calibration of 200 g F1 OIML class

| date | correction | reference | mean | std dev | UWL | LWL | UAL | LAL |
|---|---|---|---|---|---|---|---|---|
| 01/02/02 | 0,27 | 0,276 | 0,28 | 0,02 | 0,32 | 0,25 | 0,34 | 0,23 |
| 02/02/02 | 0,30 | 0,276 | 0,28 | 0,02 | 0,32 | 0,25 | 0,34 | 0,23 |
| 03/02/02 | 0,25 | 0,276 | 0,28 | 0,02 | 0,32 | 0,25 | 0,34 | 0,23 |
| 04/02/02 | 0,31 | 0,276 | 0,28 | 0,02 | 0,32 | 0,25 | 0,34 | 0,23 |
| 05/02/02 | 0,28 | 0,276 | 0,28 | 0,02 | 0,32 | 0,25 | 0,34 | 0,23 |
| 06/02/02 | 0,29 | 0,276 | 0,28 | 0,02 | 0,32 | 0,25 | 0,34 | 0,23 |
| 07/02/02 | 0,30 | 0,276 | 0,28 | 0,02 | 0,32 | 0,25 | 0,34 | 0,23 |
| 08/02/02 | 0,26 | 0,276 | 0,28 | 0,02 | 0,32 | 0,25 | 0,34 | 0,23 |
| 09/02/02 | 0,30 | 0,276 | 0,28 | 0,02 | 0,32 | 0,25 | 0,34 | 0,23 |
| 10/02/02 | 0,28 | 0,276 | 0,28 | 0,02 | 0,32 | 0,25 | 0,34 | 0,23 |




Specification limits are used to determine if the product will function in the intended fashion or if it will meet the requirements of a documentary standard. For example, we often work with weight classification schemes that provide us with specific tolerance limits.

Specification limits, are calculated as:

*Upper limit = mean or target product value + product specification limits*
*Lower imit = mean or target product value - product specification limits*

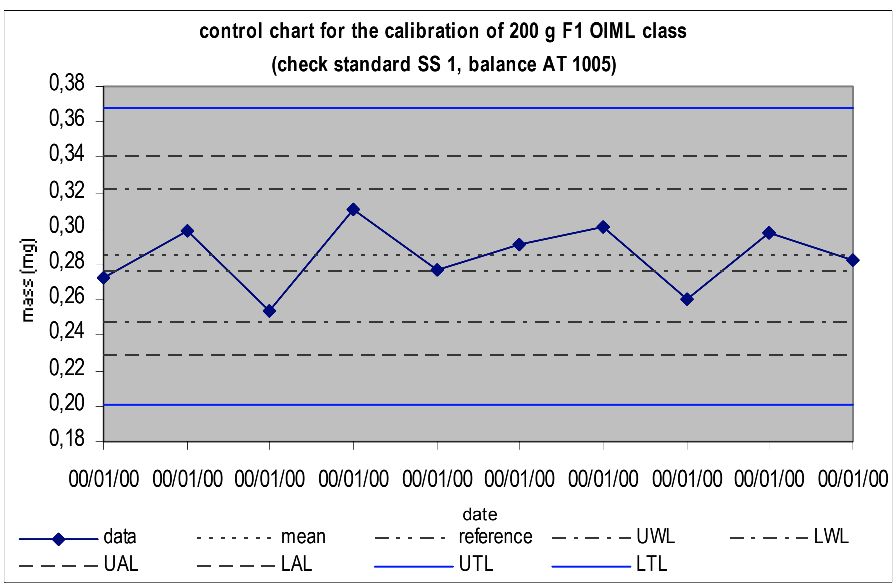
Tolerance specification limits, are calculated as:

*Upper limit = mean or nominal value + (1/3 x tolerance limit)*
*Lower limit = mean or nominal value - (1/3 x tolerance limit)*

Note: *The expanded uncertainty must be less than 1/3 of the tolerances in accordance with the classification. In practice, we may want tighter controls to ensure that the entire expanded uncertainty of each value is within the limits of 1/3 tolerance. In that case, we may want to set tolerance specification limits at 1/6 of tolerance for monitoring the measurement process.*

| | | | | |
|---|---|---|---|---|
| description | : 10 points - control chart initial data | | | |
| SOP | : direct comparison | | | |
| balance used | : AT 1005 / 0,01 mg | | | |
| load | : 200 g | | | |
| unit | : mg | | | |
| check standard | : stainless steel F1 OIML class | specification | 0,5 | mg |
| measurement process | : calibration of 200 g F1 OIML class | uncertainty tolerance / claimed uncertainty | 0,17 | mg |
| | | tolerance limit for measurement process variability | 0,08 | mg |

| date | correction | reference | mean | std dev | UWL | LWL | UAL | LAL | UTL | LTL |
|---|---|---|---|---|---|---|---|---|---|---|
| 01/02/02 | 0,27 | 0,276 | 0,28 | 0,02 | 0,32 | 0,25 | 0,34 | 0,23 | 0,37 | 0,20 |
| 02/02/02 | 0,30 | 0,276 | 0,28 | 0,02 | 0,32 | 0,25 | 0,34 | 0,23 | 0,37 | 0,20 |
| 03/02/02 | 0,25 | 0,276 | 0,28 | 0,02 | 0,32 | 0,25 | 0,34 | 0,23 | 0,37 | 0,20 |
| 04/02/02 | 0,31 | 0,276 | 0,28 | 0,02 | 0,32 | 0,25 | 0,34 | 0,23 | 0,37 | 0,20 |
| 05/02/02 | 0,28 | 0,276 | 0,28 | 0,02 | 0,32 | 0,25 | 0,34 | 0,23 | 0,37 | 0,20 |
| 06/02/02 | 0,29 | 0,276 | 0,28 | 0,02 | 0,32 | 0,25 | 0,34 | 0,23 | 0,37 | 0,20 |
| 07/02/02 | 0,30 | 0,276 | 0,28 | 0,02 | 0,32 | 0,25 | 0,34 | 0,23 | 0,37 | 0,20 |
| 08/02/02 | 0,26 | 0,276 | 0,28 | 0,02 | 0,32 | 0,25 | 0,34 | 0,23 | 0,37 | 0,20 |
| 09/02/02 | 0,30 | 0,276 | 0,28 | 0,02 | 0,32 | 0,25 | 0,34 | 0,23 | 0,37 | 0,20 |
| 10/02/02 | 0,28 | 0,276 | 0,28 | 0,02 | 0,32 | 0,25 | 0,34 | 0,23 | 0,37 | 0,20 |



## 5. USING CONTROL CHART

The overall goal in using a control chart is to efficiently ensure the quality of the measurement results by minimizing the loss of data, integrating the system into normal laboratory workload, and monitoring the standard and process for accuracy and uncertainty.

Measurement assurance should be a real time monitoring process and not one in which data are saved for entry into control charts just prior to laboratory audit or when customer calls with measurement discrepancy. Data should be plotted and evaluated immediately.

After plotting data, look for stability and randomness, be sure the latest point entered is within the established limits, or we will need to take appropriate action. Plotting real time data on the control charts can prevent the release of questionable data and possible recall of certificate for items you have already tested or calibrated.

Normal distribution is probably the most important and most frequently used distribution, both in the theory and the application of statistics. We expect our measurement data to come from a normal distribution. We looked at this distribution and considered confidence intervals and probabilities. In this chapter, think about the probability that a point will come from this distribution and be within limits on the control chart unless there is something wrong. We will consider what might be wrong and what action steps may be needed to be taken to correct the problem.

When all data incorporated on the same control chart, we can see approximately where the change occurred and predict future value, barring additional damage. The unique pattern shown on the chart is the result of a change in the value of the check standard. To conduct further analysis of these data, we could separate the before and after data and conduct *t-test* and *F-test*, or we could compare the histograms of the before and after data. The differences in the two distributions are quite obvious when they are viewed graphically.

The calculation of F-value and interpretation of F-test result can be described as follows:

$F_{value} = s_{old}^2 / s_{new}^2$ ,

$s_{old}$ is the standard deviation used to establish existing limit in the control chart, and $s_{new}$ is the standard deviation of the most recent data. Our measurement system is considered to be “out of control” if we meet the following conditions

$F_{value} > F_{v_{old}, v_{new}, \alpha}$ ; if $s_{old}^2 > s_{new}^2$ ;

$F_{value} < F_{v_{old}, v_{new}, 1-\alpha}$ ; if $s_{old}^2 < s_{new}^2$

The calculation of t-value and interpretation of t-test result can be described as follows:

$$t_{value} = \frac{\bar{x}_{old} - \bar{x}_{new}}{\sqrt{s_{old}^2 / n_{old} + s_{new}^2 / n_{new}}} ,$$

$\bar{x}_{old}$ is the mean of the old data existing in the control chart; $\bar{x}_{new}$ is the mean of most recent data; $s_{old}$ is the standard deviation used to establish existing limit in the control chart; $s_{new}$ is the standard deviation of the most recent data; $n_{old}$ the number of data points existing in the control chart; $n_{new}$ is the number of data points of most recent data. Our measurement system is considered to be “out of control” if we meet the collowing conditions:

$|t_{value}| < t_{critical}$ from the t-table

$t_{critical}$ is the critical $t_{value}$ that depends on degrees of freedom calculated using the following formula:

$$\nu = \frac{\left(s_{old}^2 / n_{old} + s_{new}^2 / n_{new}\right)^2}{\frac{\left(s_{old}^2 / n_{old}\right)}{n_{old} - 1} + \frac{\left(s_{new}^2 / n_{new}\right)}{n_{new} - 1}}$$

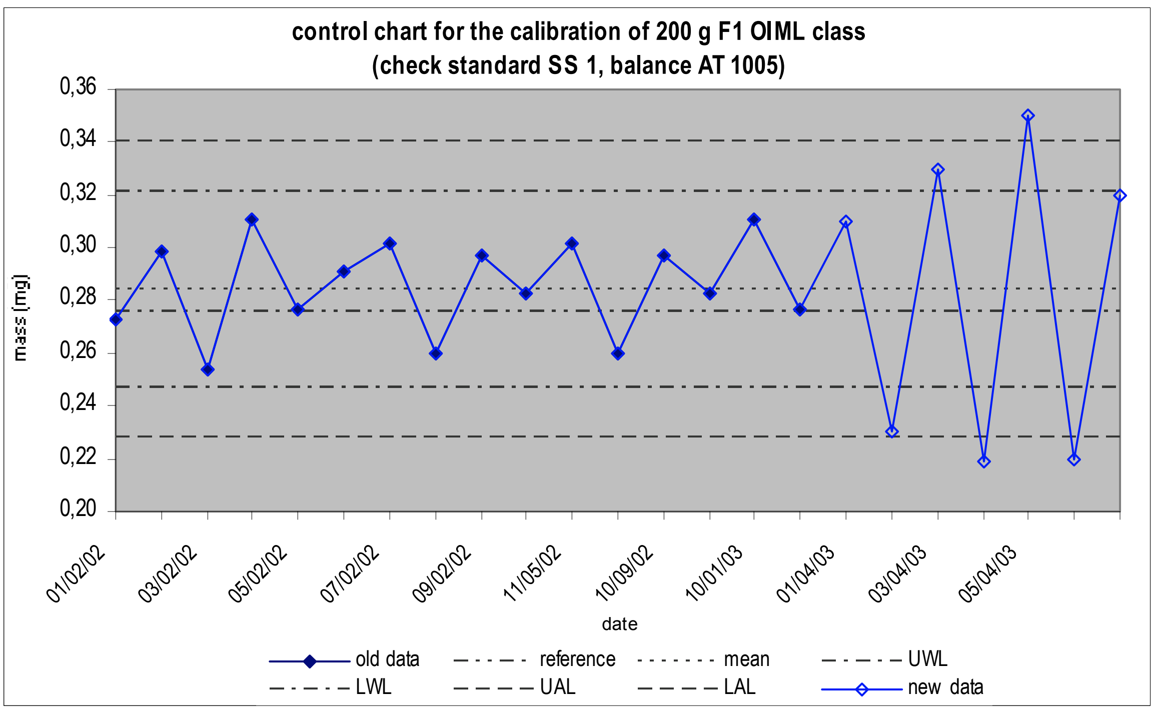
Our control charts helps to identifying stability or change in the measurement process and in the standards we need to monitor the process and the standards to ensure the quality of our measurements. In the following we identify the number of examples of "*things to look for*" in the control charts that show instability or changes:

EXAMPLE:

*CHECK and ACT*

*PROCESS DEGRADATION*

*The most recent data have wider dispersion than the earlier data on which the limits were based as shown in the following control chart.*




| OLD DATA | | NEW DATA | |
|---|---|---|---|
| Mean | 0.28 | Mean | 0.28 |
| Standard deviation | 0.02 | Standard deviation | 0.06 |
| Degrees of freedom | 15 | Degrees of freedom | 6 |
| Reference value = 0.276 | | | |
| t - value = 0.138 < 2.4 ; F – value = 0.1 < 0.358 | | | |

Possible assignable causes:

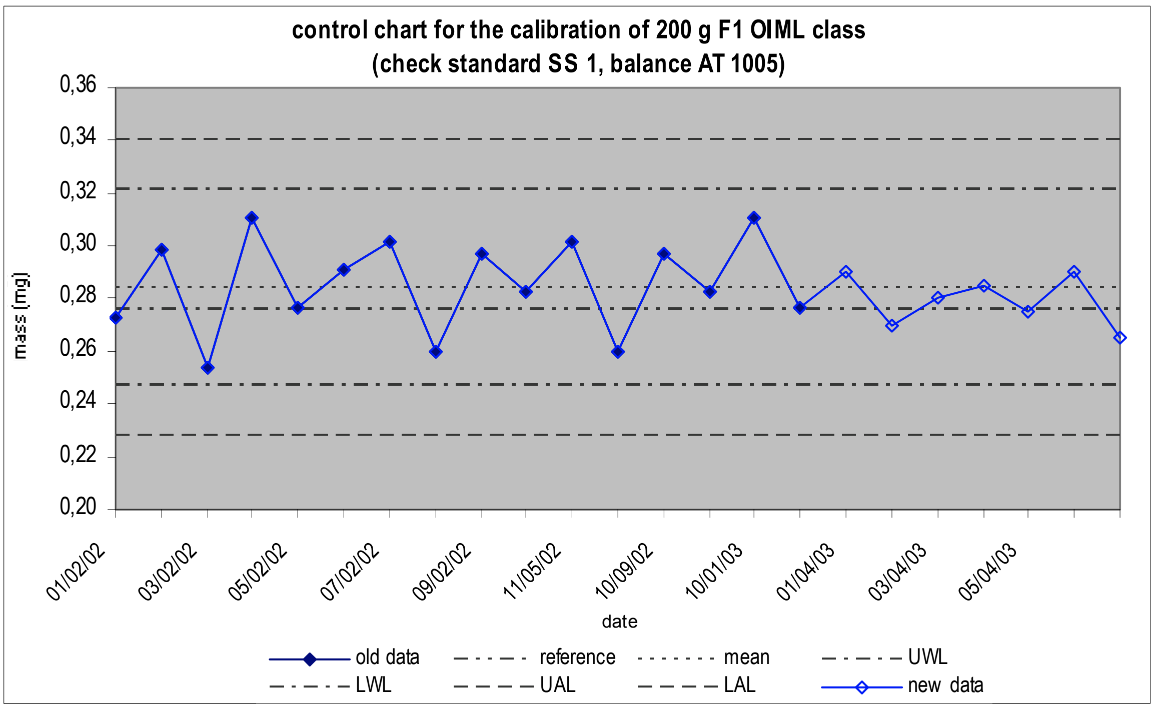
- Our balance need service
- We have new calibration officer in the laboratory who needs training
- Our environmental controls stopped working properly

Possible action steps:

- Obtain service or repair for balance
- Get new staff trained as soon as possible
- Fix environmental control as soon as possible

*PROCESS IMPROVEMENT*

*The most recent data have a narrower dispersion than the earlier data on which the limits were based as shown in the following control chart.*




| OLD DATA | | NEW DATA | |
|---|---|---|---|
| Mean | 0.28 | Mean | 0.28 |
| Standard deviation | 0.02 | Standard deviation | 0.01 |
| Degrees of freedom | 15 | Degrees of freedom | 6 |
| Reference value: 0.276 | | | |
| t - value = 1.11 < 2.09 | ; F – value = 3.4 < 3.94 | | |

Possible assignable causes:

- We obtained service for your balance
- We have a new calibration officer who has many years of previous experience
- Our environmental controls were repaired or improved

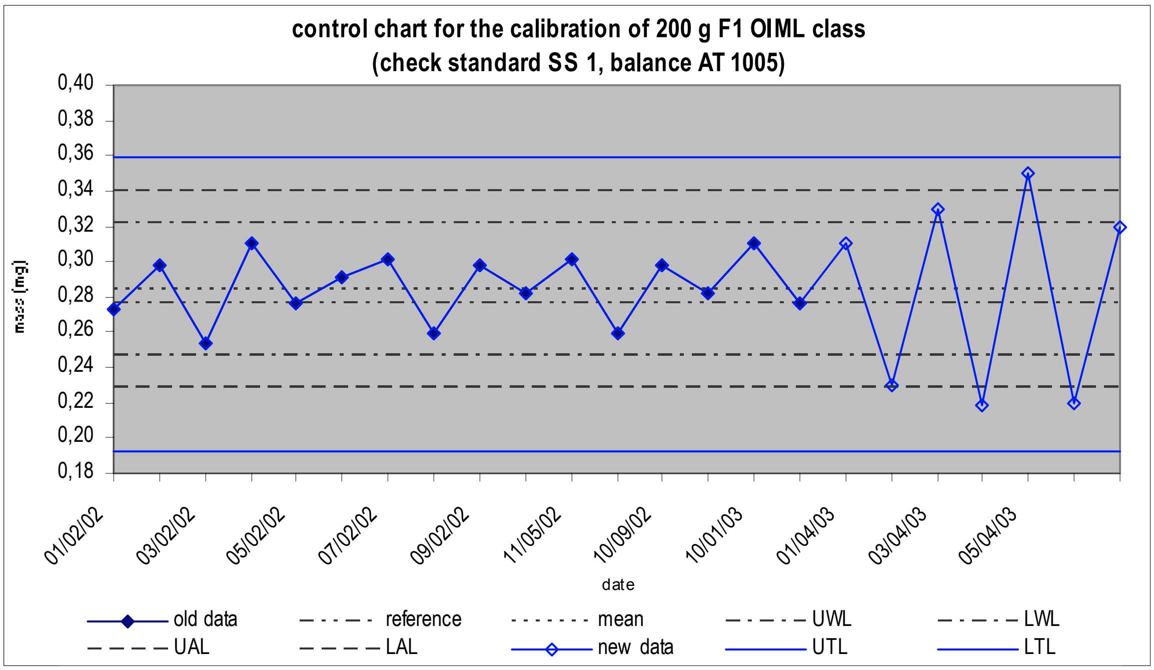
- We have been practicing in balance operation (more care in centering the weight on the pan, gently releasing the beam, allowing more stability before taking a reading)

Possible action steps:

- Recalculate your standard deviation for the measurement process and update your chart
- Recalculate tour standard deviation for the measurement process and updated your uncertainty values

*PROCESS COMPARED TO SPECIFICATION / TOLERANCES*

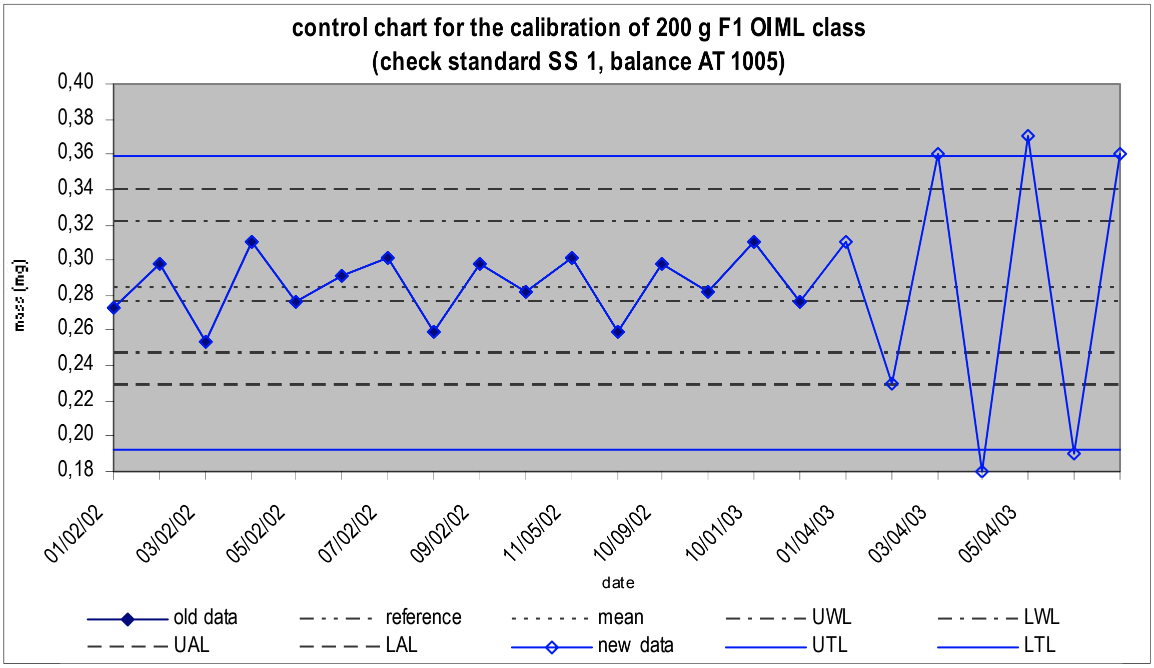
When evaluating measurement process against specifications and tolerance, we should consider whether or not the process is acceptable. If the process is acceptable, there is no need to look for assignable causes. If it is not acceptable, then we need to look for assignable causes and take action.




| OLD DATA | | NEW DATA | |
|---|---|---|---|
| Mean | 0.28 | Mean | 0.28 |
| Standard deviation | 0.02 | Standard deviation | 0.057 |
| Degrees of freedom | 15 | Degrees of freedom | 6 |
| Reference value : 0.276 | | | |
| t - value = 0.14 < 2.36 ; F – value = 0.1 < 0.358 | | | |

In the above cases, the control chart shows that the variability of the most recent data is out of the action limits and the result of F-test also shows "out of control" condition, However, the variability is still acceptable compared the tolerance limits, then there is no need to look for possible assignable causes if we used the tolerance limits as a action limits for our measurement processes.

If our control chart shows that the variability of most recent data exceeds tolerance limits as shown in the following control chart:




| OLD DATA | | NEW DATA | |
|---|---|---|---|
| Mean | 0.28 | Mean | 0.28 |
| Standard deviation | 0.02 | Standard deviation | 0.08 |
| Degrees of freedom | 15 | Degrees of freedom | 6 |
| Reference value : 0.276 | | | |
| t - value = 0.001 < 2.36 ; F – value = 0.04 < 0.358 | | | |

Possible assignable causes:

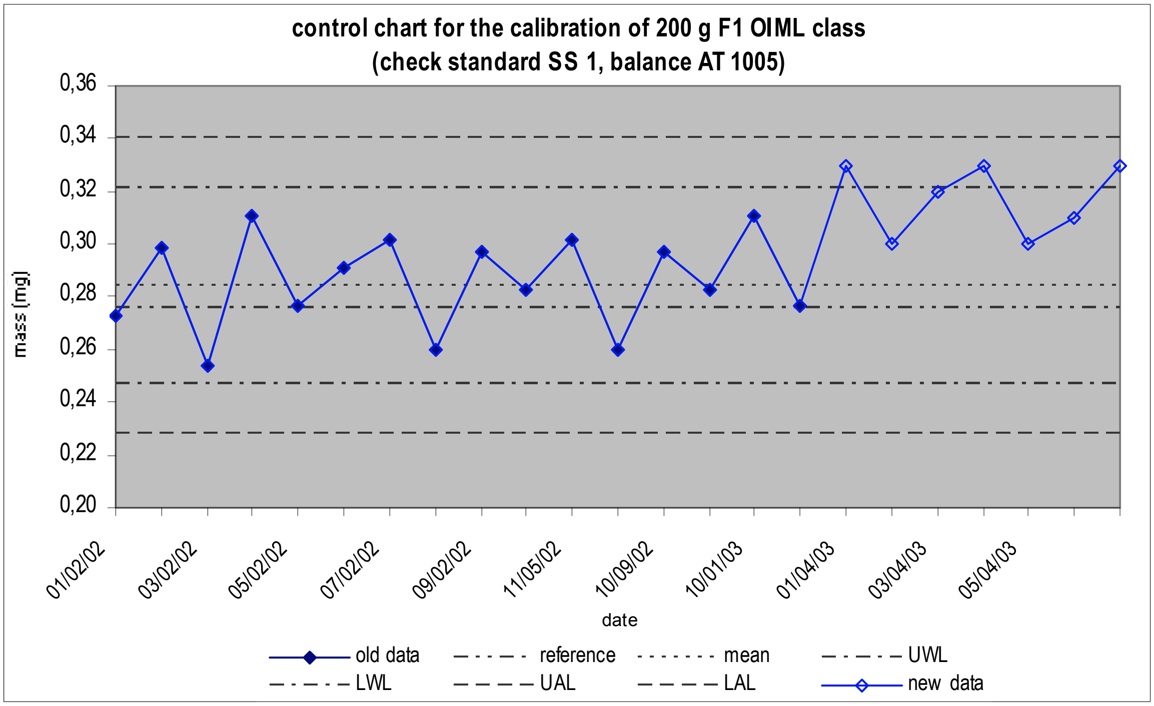
- Our process is not achieving the requirements needed to meet the tolerances because our balances has degraded and can not reach its normal performance
- The process may be acceptable for the clients needs even though it is larger than the tolerances we have assigned to the process

Possible action steps:

- Select or purchase a better balance that will meet the needs of our clients and/or our stated tolerance
- Talk with our client to determine real, rather than stated measurement requirements

SHIFT IN VALUES OF STANDARD

All of the most recent data are above the mean value of the earlier data as shown in the following control chart:




| OLD DATA | | NEW DATA | |
|---|---|---|---|
| Mean | 0.28 | Mean | 0.32 |
| Standard deviation | 0.02 | Standard deviation | 0.014 |
| Degrees of freedom | 15 | Degrees of freedom | 6 |
| Reference value : 0.276 | | | |
| t - value = 4.54 > 2.13 ; F – value = 1.7 < 3.94 | | | |

Possible assignable causes:

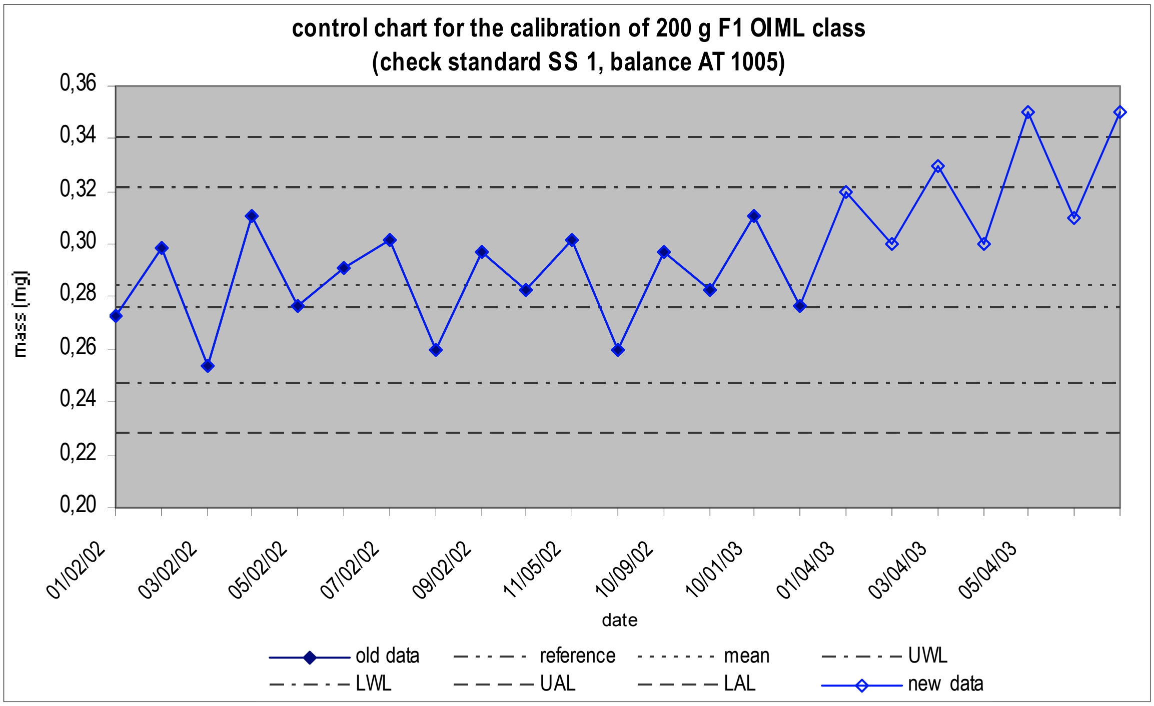
- Our standard is damage
- Our check standard is damage
- We are using calculation software that has not been validated
- We have errors in entering data and not to double check data
- Our equation for the correction due to environmental condition were drifted and correction changed
- Our standard was recalibrated and a new value was assigned without starting a new control chart

Possible action steps:

- Correct for each of the above causes
- For damage to standard and check standard, a recalibration of the standards is not enough, you must determine how and why the standard was damage and make sure that it does not happen again

DRIFT OF VALUES OF STANDARD

The recent values seem to be steadily increasing above the previous mean values as shown in the following control chart:




| OLD DATA | | NEW DATA | |
|---|---|---|---|
| Mean | 0.28 | Mean | 0.32 |
| Standard deviation | 0.02 | Standard deviation | 0.021 |
| Degrees of freedom | 15 | Degrees of freedom | 6 |
| Reference value : 0.276 | | | |
| t - value = 4.00 > 2.23 ; F – value = 0.72 > 0.36 | | | |

Remember that the stability is less important than the predictability. In fact, some laboratories apply correction to their check standard based on a consistent history of drift, they have a level of confidence for predicting drift and incorporate an uncertainty associated with the predicted correction.

Possible assignable causes:

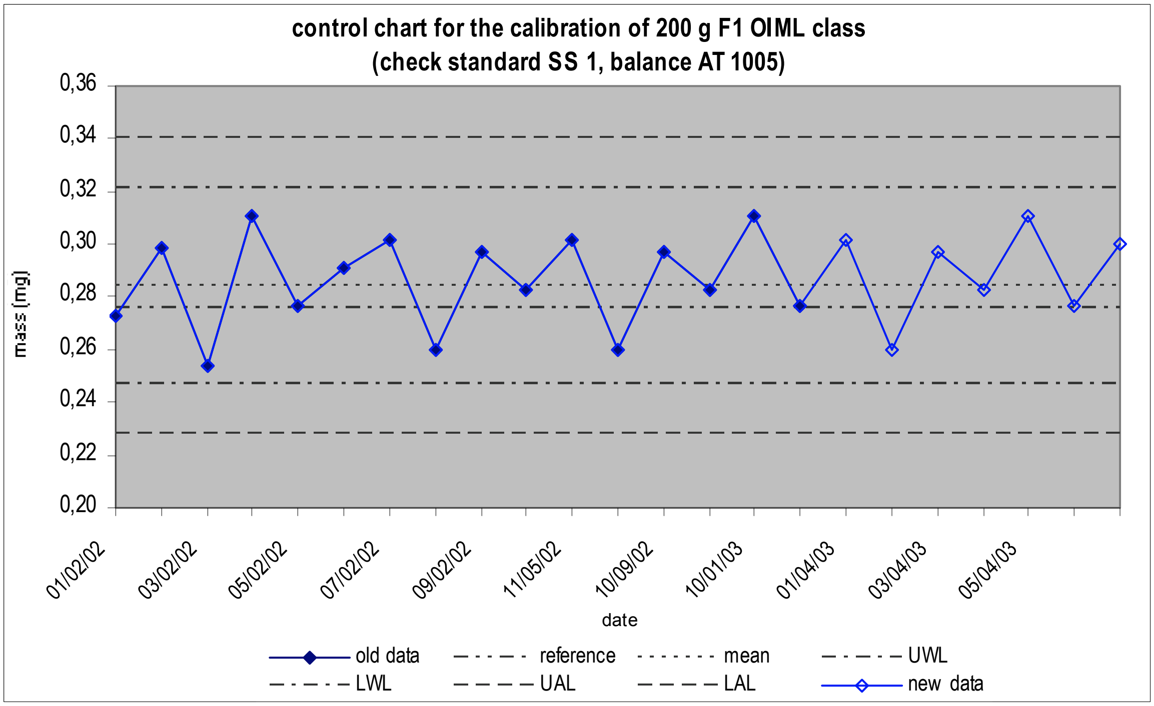
- Our working standard worn from use
- Our check standard getting dirty
- Our standards are not stable enough for this process

Possible action steps:

- Evaluate drift rate of standard, which may be used to evaluate calibration intervals
- Incorporate the level of offset at some future points in time
- Include the uncertainty of the offset from reference values now and in the future

BIAS OF STANDARD COMPARED TO REFERENCE

In our example, there is difference between the mean value and the reference value obtained from the independent calibration of check standard as shown in the following control chart:




| OLD DATA | | NEW DATA | |
|---|---|---|---|
| Mean | 0.28 | Mean | 0.28 |
| Standard deviation | 0.02 | Standard deviation | 0.018 |
| Degrees of freedom | 15 | Degrees of freedom | 6 |
| Reference value : 0.276 | | | |
| t - value = 0.497 < 2.18 ; F – value = 1.06 < 3.94 | | | |

Possible assignable causes:

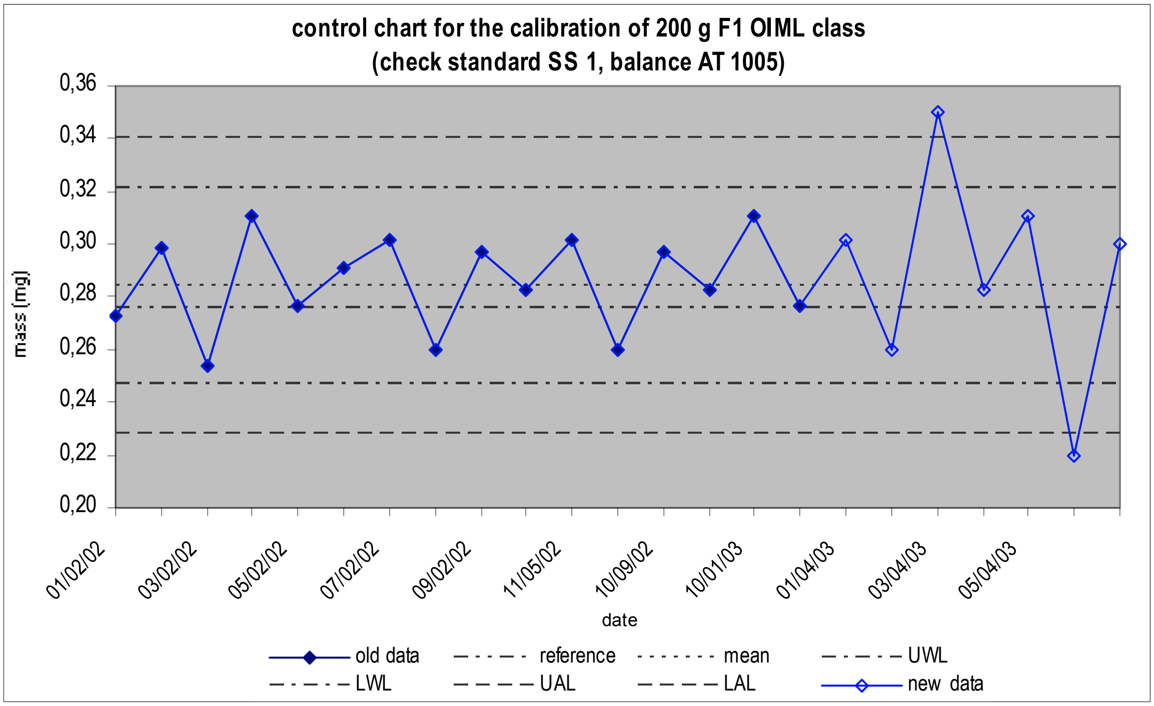
- Repeated observation simply shows a measurement bias that is within the uncertainty of calibrated values
- An error in the density of standard that affect buoyancy correction
- An error of environment correction equipment causing bias
- Standard changed at some point in the past and the shift is not reflect the current chart
- It may be using standard operating procedure that does not compensate for drift

Possible action steps:

- Evaluate bias with respect to the uncertainty and if it is small enough, incorporate as an uncorrected systematic error
- Obtain a calibration for standard, check standard and environmental equipment
- Get density measurement for our standard or check standard
- Use standard operating procedure that compensate for drift

OUTLIERS

There are a few of data from a recent measurement fall outside the action limits as shown in the following control chart:




| OLD DATA | | NEW DATA | |
|---|---|---|---|
| Mean | 0.28 | Mean | 0.29 |
| Standard deviation | 0.02 | Standard deviation | 0.041 |
| Degrees of freedom | 15 | Degrees of freedom | 6 |
| Reference value: 0.276 | | | |
| t - value = 0.214 < 0.236 ; F – value = 0.19 < 0.36 | | | |

If we can identify the cause of outliers and take appropriate action, we can make a note of the action in our control database and flag the points so they are not included in the statistical calculations. Do not delete these points from control database, if we find problems that cause outliers every 10$^{th}$ data point, it means the corrective action steps are not effective. If we arbitrarily delete observations, we will not be able to track erratic readings.

Possible assignable causes:

- Balance not exercised normally / properly
- We do not allow adequate thermal equilibrium to the standard prior to begin calibration
- We have data entry error or miscalculation
- Our environmental system is out of control
- Our standard was recently damaged and this is the first indication of problem (we would only see the data “out” on one side of the chart, if were damaged, which is not the case here)

Possible action steps:

- Immediately make another measurement to check the validity of the current measurement result and then make a determination about what to do
- Another means for calculating the data may be used if an error was found

## 6. SOURCES OF MEASUREMENT DATA FOR MEASUREMENT ASSURANCE

Every laboratory has a number of measurement processes that should be identified during the planning stage of PLAN – DO – CHECK – ACT cycle. Each measurement process produces unique data that we can use to assure the quality of our measurements. Each of these sources of data may be plotted, tracked and/or controlled using control chart technique

It is important to integrate the analysis of our data sources and consider whether data from one process is related to another, and whether one set of data supports our assessment and tentative conclusions regarding another set of data. All of our data sources are unique but they fit together like pieces of a puzzle. The sources of measurement data we can used to monitor measurement process may include, but not limited to:

a. <u>Calibration of reference standards</u>
   - We can check the values for our reference standards over time
   - The stability of reference standards is the main thing we should look for because the values may be determined using different processes and different laboratories over a long period of time. This is unlike tracking the values for our check standards, because tracking the checks standard values represents a process that we control within our laboratory. We don  not always control the process for the calibration of our standards
   - By analyzing drift rates (due to possible wear) and variability (stability) of our standards, we can determine suitable calibration intervals for our standards. We can also determine if a change occurs

b. <u>Proficiency testing data</u>
   - Data obtain for proficiency testing by inter-laboratory comparison often reflect characteristics common to data from control charts for other standards and/or measuring equipments maintained in the laboratory. Bias that is observed in a proficiency testing results may also be evident in a control chart that monitor the standards and/or measuring equipments that were used for particular proficiency testing program
   - For example: we have participated in inter-laboratory comparison  with a 200 g mass standard using substitution procedure, and our results showed a bias of 0.68 mg compared with the accepted reference value. We take a look at the chart we have for the calibration history of our working standard and note that we have a noticeable drift in mass values and that the calibration interval was nearly exceeded. Then, we estimate the drift rate and find that the mass value for our working standard is likely off by 0.60 mg to 0.70 mg in the same direction as the bias observed in the inter-laboratory comparison. The values from two sources agree and we need to calibrate our working standard

c. <u>Intermediate check</u>
   - Intermediate check is the checking of our standards and/or measuring equipments between official calibrations scheduled in the calibration program, to provide additional assurance that all values continue to be stable. It provides an intermediate verification of the values for our standards and/or measuring equipments
   - Intermediate check may be conducted that are internal to our laboratory but which are used with limited frequency, perhaps only for this purpose intermediate check may also be conducted with

standards that are calibrated outside our laboratory and provided or obtained specialty for the purpose of intermediate check of our standards and/or measuring equipments

d. Calibration of working standards or measuring equipments
   - We can track the values of working standards and/or measuring equipments over time. We will look for the values and we may also be able to analyze different process by tracking the procedures that have been used in the calibration of working standards and/or measuring equipments over a long time
   - By analyzing drift rates (due to possible wear) and variability (stability) of our working standards and/or measuring equipments, we can determine appropriate calibration intervals for working standards and/or measuring equipments (just as we can do with our reference standards)
   - We can also determine if a change in the values of our working standards and/or measuring equipments occurs if measurement assurance program for our routine calibrations and/or testing are modeled to monitor our working standards and/or measuring equipments, then our charts provide warning signs for recalibration

e. Calibration or testing of customer item
   - Every process used to calibrate and/or test customer items should have a measurement assurance system applied. A check standard is needed to reflect the standard deviation of the process and the validity of the standards and/or measuring equipments on the date of the test
   - However, as you have seen, calibration and/or testing of customer items is not the only measurement process in our laboratory where measurement assurance methodologies are useful

## 7. REFERENCES

1. Harris G. L, "*NIST Special Publication 1001: Basic Mass Metrology CD ROM*", National Institutes of Standards and Technologies, Technology Administration, US Department of Commerce, September 2003
2. NIST/SEMATECH, "*Engineering Statistics Handbook*", electronic file web based handbook at http://www.itl.nist.gov/div898/handbook/, National Institutes of Standards and Technology, Technology Administration, US Department of Commerce, November 2003