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Selected Laboratory and Measurement Practices and Procedures to Support Basic Mass Calibrations (2014 Ed)

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Office of Weights and Measures
Physical Measurement Laboratory

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U.S. Department of Commerce
Penny Pritzker, Secretary

National Institute of Standards and Technology
Willie May, Acting Under Secretary of Commerce for Standards and Technology and Acting Director

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Foreword

This NIST IR of Selected Publications was been compiled as an interim update for a number of Good Laboratory Practices, Good Measurement Practices, Standard Operating Procedures, Statistical Techniques and Reference Tables. This interim publication was used as a reference document for the CD-ROM Mass Metrology Course (SP 1001) prepared by the NIST Office of Weights and Measures in 2003 and was updated again in 2012.

Many of these procedures are updates to procedures that were originally published in NBS Handbook 145, Handbook for the Quality Assurance of Metrological Measurements, in 1986, by Henry V. Oppermann and John K. Taylor. The 2003 updates incorporated many of the requirements noted for procedures in ISO Guide 25, ANSI/NCSL Z 540-1-1994, and ISO/IEC 17025 laboratory quality systems. The major changes incorporated 1) uncertainty analyses that comply with current international methods and 2) measurement assurance techniques using check standards. No substantive changes were made to core measurement processes or equations, with the exception of SOP 2, Standard Operating Procedure for Applying Air Buoyancy Corrections. Procedures were updated in 2003 in conjunction with Jose A. Torres-Ferrer.

The CIPM 1981/91 equation for calculating air density was added to SOP 2 in 2003 and updated in 2012 based on the 2007 CIPM equation.

The following Practices and Procedures were new in the 2012 publication:
Good Measurement Practices for:

- Assignment and Adjustment of Calibration Intervals for Laboratory Standards (11)
- Standard Operating Procedure Selection (12)
- Ensuring Traceability (13)
- Selection and Use of Sensitivity Weights in Weighing Procedures (14)

Standard Operating Procedures for:

- Assignment of Uncertainty (29)
- Process Measurement Assurance Program (30)
- Standard Operating Procedure for the Calibration of Weight Carts (33)

The essential portions of SOP 29, Standard Operating Procedure for Assignment of Uncertainty were first published in 1994 for the NCSL Conference and Symposium and to assist State weights and measures laboratories comply with the ISO Guide to the Expression of Uncertainty in Measurements (1993).

Key updates in 2012 were made to address updated definitions of metrological traceability. Key updates in 2014 have been to clarify procedures after national review by users as well as to add example uncertainty budget tables in each SOP.

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Good Laboratory Practices

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GLP 1

Good Laboratory Practice for the Quality Assurance of the Measurement Process[1]

Quality Assurance of the Measurement Process means establishing, documenting, implementing, and maintaining a quality system appropriate to the laboratory's scope of activities. Having such a system in place will allow the laboratory to know, within the limits of a measurement process, that a measurement is valid with respect to its traceability, accuracy, and precision.

The validity of tests and calibrations should be monitored with quality control procedures. Statistical techniques are used to record and monitor charted measurement results to permit the detection of trends. The metrologists and laboratory management should plan and review the results from quality assurance monitoring.

Other steps taken to ensure the quality of the measurement process may include, but are not limited to:

- the regular use of Standard Reference Materials (SRMs) and /or internal quality control using secondary reference materials;
- participation in interlaboratory comparisons (round robins);
- test replications with same and/or different methods;
- recalibration of retained items;
- correlation of different characteristics of an item; and
- proper calibration intervals.

Each measurement parameter in the laboratory's scope of activities must be reviewed and analyzed to determine the validity of the measurement process.

The standards and the measurement process for each parameter must be in a state of statistical control. Statistical control means that the variability of the measurement process is known and stable; when a process is in statistical control, we can assume that the reported measurement uncertainties are valid. The National Institute of Standards and Technology provides technical guidance and support to laboratories to develop suitable measurement control programs that provide measurement assurance. The objective of these programs is to evaluate the entire measurement process including:

- procedures;
- standards;
- equipment;
- personnel; and
- environment.

[1] See also Section 5.9 of ISO/IEC 17025:2005 (reviewed 2010) General Requirements for the Competence of Testing and Calibration Laboratories.

While other quality assurance programs could meet these objectives, the control programs developed for measurement assurance greatly increase the comprehensiveness of the program.

State weights and measures laboratories typically provide measurement services in the disciplines of mass, volume, and length. Some of the weights and measures laboratories provide services in other measurement areas. Approximately 89 % of their workload is in mass standards calibration.[2] Mass calibration is the first discipline for which a measurement assurance program was developed in the 1960's and was implemented in State laboratories in the 1980's. Nevertheless, all measurement disciplines must have a measurement assurance system in place.

The most recent improvement in assuring the quality of each measurement parameter in the State Laboratories is the incorporation of a Process Measurement Assurance Program (PMAP).[3]

The PMAP system consists of duplicating the measurement process by including a check/control standard as surrogate for the test item. Measurements made over an extended period of time, typically at least a year, will show all the conditions that are likely to affect the measurement process and their combined effects. Controlled duplication of the measurement process provides for the realistic evaluation of the measurement variability as one of the primary components in the estimation of the measurement uncertainty.

Measurement results that are collected over several years may be statistically evaluated with current results being compared to results from previous years. Any observed problems or changes in the measurement results are investigated and if necessary, corrective action can be taken. Ongoing monitoring establishes a continuous, comprehensive, internal measurement assurance program in the laboratory.

Data from internal measurement assurance programs may be compared to the results of inter-laboratory comparisons (round robins) or proficiency tests in which the laboratory participates.

The strength of the measurement assurance approach lies in its applicability to a wide variety of measurements with sufficient flexibility to permit each measurement control program to be tailored to the particular needs of a given measurement area. The sophistication of the control program depends on the criticality of the measurement.

[2] State Laboratory Program Workload Survey, 2011, NIST/OWM and NCSLI Legal Metrology Committee.
[3] See NISTIR 6176 (1998) and NISTIR 6969, SOP 30 (2014).

GLP 4

Good Laboratory Practice
for
Periodic Recalibration of State Standards

A number of States have provisions in their weights and measures laws that require the periodic submission of their State standards to NIST for calibration. Those provisions are based on an early version of the Model Law (1911), which was considered appropriate for the circumstances that prevailed prior to the establishment of the New State Standards Program by NIST. Periodic calibration is necessary on a defined regular, periodic basis, and also when measurement control results from internal control charts or external round robins indicate questionable data.

Standards of mass, volume, and length, fabricated from modern materials, kept in the controlled environment of a State metrology laboratory under the custody of trained metrologists, are generally stable and not subject to excessive change. The cooperative NIST-State audit programs often identify changes in ample time for corrective action in the unlikely event that such a change should occur. These same programs provide the necessary evidence of the ability to provide metrological traceability of measurement results at a level of confidence sufficient for the need.

The process of packing, shipping, and unpacking exposes the standards to unnecessary hazards that could result in damage, compromising their integrity. The return and re-calibration could take several months causing an unavailability of State services that would be disruptive to the performance of the mission of the State laboratories.

In order to develop a policy for the guidance of and implementation by all 50 States regarding this subject, the following actions are recommended:

1. States should recognize the fact that periodic return of their State standards to NIST (or another suitable and accredited calibration laboratory) for re-calibration is essential to comply with State laws regarding traceability and with ISO/IEC 17025, GMP 11, and when: 1) data regarding traceability is unavailable; 2) charted measurement results indicate that the standards are out of control; 3) measurement results on round robins or performance tests are suspect; or 4) NIST advises the State of the need.

2. GMP 11 and GMP 13 provide the basis for documenting metrological traceability and baseline calibration intervals. These publications are templates that must be modified to specific measurement parameters and applications in each laboratory. Documented calibration intervals may state that calibrations are obtained whenever evidence from outside evaluations, proficiency tests, and/or internal controls indicate that significant changes may have taken place, or when measurement results are suspect, but shall not exceed a specified number of years (with the number specified). Statements such a “calibration as needed” are unacceptable.

3. References to the periodic re-calibration of State standards in the law such as,

   "He (the director) shall maintain the State standards in good order and shall submit them, at least once in ten years, to the National Institute of Standards and Technology for calibration,"

   should be followed if present and may be amended to reflect a more frequent interval, plus calibration based on identified needs. Alternatively, the wording of Sections 3 and 12 of the present Uniform Law from NIST Handbook 130[1] may be substituted:

   SECTION 3. PHYSICAL STANDARDS. -- Weights and measures that are traceable to the U.S. prototype standards supplied by the Federal Government, or approved as being satisfactory by NIST, shall be the state reference and working standards of weights and measures, and shall be maintained in such calibration as prescribed by the NIST as demonstrated through laboratory accreditation or recognition. All field standards may be prescribed by the Director and shall be verified upon their initial receipt, and as often thereafter as deemed necessary by the Director.

   SECTION 12. POWERS AND DUTIES OF THE DIRECTOR. -- The director shall:

   12 (a) maintain traceability of the state standards as demonstrated through laboratory accreditation or recognition.

The approach described above is recommended by NIST because each State that participates in the Weights and Measures Division Laboratory Recognition Program has the capability of evaluating its own State standards with the necessary documentation referencing the national standards. The Laboratory Recognition Program provides interaction between the State standards laboratories and NIST, ensuring satisfactory laboratory conditions suitable for primary standards in addition to the proper use of NIST procedures in standards calibration. Thus, each State program is evaluated and, if found in compliance, is Recognized as being capable of performing the measurements listed on the Certificate of Measurement Traceability.

[1] NIST Handbook 130, Uniform Laws and Regulations in the Areas of Legal Metrology and Engine Fuel Quality (Latest Edition). This text was originally published in the 2005 version of Handbook 130.

GLP 9

Good Laboratory Practice for Rounding Expanded Uncertainties and Calibration Values

A calibration is not complete until the expanded uncertainty associated with the calibration is determined and reported. Each Standard Operating Procedure (SOP) includes information regarding the calculation of uncertainties. The expanded uncertainty is generally reported with approximately a 95 % confidence interval. The confidence interval is determined by multiplying a coverage factor (often 2, but based on the degrees of freedom or effective degrees of freedom) times the root sum square of properly combined Type A and Type B evaluated components according to the ISO/IEC Guide to the Expression of Uncertainty in Measurement (GUM, 2008), Section 7, and in ISO 80000-1:2009, *Quantities and units, Part 1*.

The steps for reporting corrections and uncertainties are as follows:

1. Round the uncertainty to two significant figures following the rules given here.
2. Round the correction/error to the last figure affected by the uncertainty.
3. Report the rounded correction value and uncertainty to the same level of significance.

Rounding Rules

1. Identify the first two significant digits. Moving from left to right, the first non-zero number is considered the first significant digit. Zeros, which follow a decimal point, when there are only zeros ahead of the decimal point, are not considered significant figures.

2. Use the following rules to round measurement data, consistent with its significance:

    1. When the digit next beyond the one to be retained is less than five, keep the retained figure unchanged. For example: 2.541 becomes 2.5 to two significant figures.

    2. When the digit next beyond the one to be retained is greater than five, increase the retained figure by one. For example: 2.453 becomes 2.5 to two significant figures.

    3. When the digit next beyond the one to be retained is exactly five, and the retained digit is even, leave it unchanged; conversely if the digit is odd, increase the retained figure by one (even/odd rounding). Thus, 3.450 becomes 3.4 but 3.550 becomes 3.6 to two significant figures.

    Note: Even/odd rounding of numbers provides a more balanced distribution of results. Use of computer spreadsheets to reduce data typically follows the practice of rounding up.

4. When two or more figures are to the right of the last figure to be retained, consider them as a group in rounding decisions. Thus, in 2.4(501), the group (501) is considered to be greater than 5 while for 2.5(499), (499) is considered to be less than 5.

Several examples to illustrate the proper method of reporting corrections and uncertainties follow.

Example 1

Suppose the correction for a weight is computed to be 1.357 8 mg and the uncertainty is 0.577 5 mg. First, round the uncertainty to two significant figures, that is, 0.58 mg. Then state the correction as 1.36 mg. Notice that the uncertainty and the correction express the same number of decimal places. Report the correction as 1.36 mg ± 0.58 mg.

Example 2

The volume of a given flask is computed to be 2000.714 431 mL and the uncertainty is 0.084 024 mL. First, round the uncertainty to two significant figures, that is, 0.084 mL. (Do not count the first zero after the decimal point.) Round the calculated volume to the same number of decimal places as the uncertainty statement, that is, 2000.714 mL. Report the volume as 2000.714 mL ± 0.084 mL.

Example 3

The correction for a weight is computed to be 4.3415 mg and the uncertainty is 2.0478 mg. First, round the uncertainty to two significant figures, that is, 2.0 mg. (Notice that two significant figures are shown. The zero is a significant figure since it follows a non-zero number.) Then, round the correction to the same number of decimal places as the uncertainty statement, that is, 4.3 mg. Report the correction as 4.3 mg ± 2.0 mg.

Example 4

The correction for a weight is computed to be 285.41 mg and the uncertainty is 102.98 mg. Because this uncertainty is a large number, we first convert both values to the next larger commonly reported unit (i.e., 0.28541 g and 0.10298 g respectively). First, round the uncertainty to 0.10 g. (The first nonzero digit (1) is the first significant figure and the remaining digits are rounded to the nearest number following the first nonzero digit.) Then, round the correction to the point where the rounding occurred in the uncertainty statement. Round the correction to 0.29 g. Report the correction as 0.29 g ± 0.10 g.

Example 5

The correction for a weight is computed to be 285.41 mg and the uncertainty is 33.4875 mg. First, round the uncertainty to two significant figures, that is 33 mg. Then, round the correction

to the same number of decimal places as the uncertainty statement, that is, 285 mg. Report the correction as 285 mg ± 33 mg.

Example 6

The length of a calibrated interval is computed to be 9.999 455 8 ft and the uncertainty is 0.003 561 7 in. First, make sure both values are reported in the same unit (i.e., convert the uncertainty to ft, 0.000 296 808 ft.) Then, round the value to two significant figures, that is, 0.000 30 ft. Then, round the length of the interval to the same number of decimal places as the uncertainty value, that is, 9.999 46 ft. Report the length of the interval as 9.999 46 ft ± 0.000 30 ft.

Rather than stating the uncertainty value with each correction, it is also proper to place the correction values in a column headed by the words "Correction" or "Error," etc., and place the uncertainties (without plus or minus signs) in a column headed "Expanded Uncertainty".

GLP 11

Good Laboratory Practice for Painting Cast Iron Weights

Large cast iron or steel weights should be painted both for their protection and to preserve their mass integrity. Unprotected weights are subject to corrosion. Furthermore, when corrosion is present, the extent and any changes resulting from it may be difficult to estimate.

Thin even coats of aluminum paint are recommended for this purpose. Spray applications are best if large weights or a number of small weights are to be painted. In preparation for painting, a weight should be cleaned and loose scale should be removed using a wire brush (DO NOT remove old paint and corrosion by means of sand blasting or pressure washing. Severe damage may result.). The painting should be done before the weights are calibrated or tolerance tested, unless arrangements are made to obtain "as found" values. If “as found” values are desired, two tests may be required.

Painting should be done in an area reserved for this purpose, or at least in a place which is removed from laboratory measurement operations. Painting operations must be in compliance with applicable safety standards. The weights should be protected from dust or dirt contamination while the coating is drying.

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Good Measurement Practices

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GMP 1

Good Measurement Practice for Reading Turning Points on an Equal-Arm Balance

The damping of the beam oscillations of an equal-arm balance is generally very slow. Consequently, it is practical to read the turning points (the highest and lowest value of the beam oscillation) on the graduated scale and use the sum of the turning points as the observation value rather than wait for the beam to come to rest. This GMP requires that the graduated scale be numbered such that adding weights to the left arm increases the readings.

Suppose the graduated scale has 20 graduations that are numbered from 0 to 20. The loads on the balance arms should be adjusted so that the sum of the turning points is approximately twice the midscale reading. In this example, the sum of the turning points should be within one division of twenty. Turning points should be estimated to at least 0.1 division in this example, which is typical of the Russell balance. This means that the final rest point is approximately 10, the midscale reading. Motion to the beam may be induced or dampened so that the turning points can be read easily. Care should be taken so that the beam does not hit limiting stops during its normal oscillation while turning points are being read. The amount of the beam oscillation is not critical although a span of from three to ten divisions is adequate.

Once motion has been induced for the beam oscillation, wait for at least one complete oscillation cycle for the beam motion to stabilize. After this time, the turning points can be read. The readings may begin with either the high or low turning point. The turning points for at least two consecutive oscillation cycles should be recorded. The turning points should reveal a consistent pattern of slow damping; that is, the turning points should gradually converge to the eventual rest point. For example, if the last high turning point was greater than the previous high turning point (assuming that the readings normally drop on successive readings), this would indicate that something has interfered with beam oscillation, hence the last reading was not valid with respect to the previous readings. Under these circumstances, turning points should continue to be read until a consistent damping pattern has been obtained. In some cases, the balance oscillation will dampen so slowly that the same readings may be obtained for several oscillations before a decrease is observed. These readings are valid and may be used to compute the sum of the turning points.

When at least four satisfactory turning points have been obtained (two high and two low turning points), all but the last three readings should be discarded. This will leave two high and one low turning point or vice versa. <u>The two readings for the high or low turning points, as the case may be, should be averaged and added to the single turning point to obtain the sum of the turning points</u>. The sum should be carried out to two decimal places if the second decimal place digit is nonzero. As an example, the following readings were obtained: 15.5, 4.3, 15.4, 4.4. The sum of the turning points is computed as $\left(\frac{4.3+4.4}{2}\right)+15.4=19.75$.

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GMP 10

Good Measurement Practice for Understanding Factors Affecting Weighing Operations

Good laboratory techniques and practices, as well as good standards and balances, are required to obtain good mass measurements. Errors can arise from a variety of sources, such as balance drift, air currents, lack of thermal equilibrium, magnetic or electrostatic fields, and to manipulative skills using the balance. The magnitude and significance of these errors depends upon the laboratory environment, the balance, and the accuracy required for the measurement. Different methods of weighing are used to eliminate or reduce the effects of sources of error. Particular sources of error and ways to eliminate the errors are discussed with the weighing procedures. The accuracy required for a measurement and the criticality of the measurement often influence the choice of the weighing method used in the measurement. Regardless of the procedure used, several practices should be followed to promote good measurements.

Environment and Preparation

First, the balance should be installed in a laboratory having good temperature and humidity control. The requirements for temperature stability increase as more precision is needed in the measurement. For high precision measurement, the temperature should not vary by more than ± 1 °C in a 24 h period throughout the year and should vary by less than 0.5 °C during the course of any one measurement series (typically less than 1 h). General laboratory measurements can probably withstand temperature variations of ± 2 °C per day. Varying temperatures result in balance drift and in unequal temperatures for the air, balance, and objects to be weighed. The relative humidity should be maintained between 40 % and 60 % which is the recommended limits provided by most balance manufacturers and by OIML R 111. If the relative humidity falls significantly below 40 %, electrostatic discharge may be generated both in and near the balance. This can result in erratic readings and make precision weighing impossible. If precision mass standards are cleaned, they should stabilize in the laboratory environment for at least seven to ten days.

Thermal and environmental equilibrium are critical for mass calibrations. This is why each mass SOP will provide instruction that the environment must be stable and the weights set in or near the balance for 24 hours prior to a calibration. The test objects, balances, reference standards, and air should all be in thermal equilibrium. When possible, the objects to be weighed and the reference standards should be placed either in the balance chamber or adjacent to the balance so all can come to the same thermal equilibrium. If there is a lack of thermal equilibrium, convection currents will be generated when an object is on the balance pan and an erroneous reading may result. These types of errors are likely to go unnoticed when check standards are handled the same way, so care must be taken to reduce the likelihood of their occurrence. Tests have shown that these types of errors depend upon the temperature differences among the objects and the balance, and on the geometry of the objects being weighed. On 20 g standards of greatly differing geometry, studies have shown that errors as large as 0.2 mg can occur for a 4 °C temperature difference among the standards and the balance. Errors as large as 3 mg have also

been observed at 1 kg loads when standards were stored in a cabinet and unknown test items near the balance where a temperature gradient of 2 °C was present (when the uncertainty was less than 0.1 mg). When weights are delivered to the laboratory, care must be taken to ensure adequate environmental equilibration, especially when outside conditions deviate from those in the laboratory by more than 10 °C. The presence of frost, ice, or condensation on the weights is a common occurrence and may not be readily visible.

The balance must be installed in an area free from air currents. Balances should be installed away from heating/cooling vents. It is not adequate to merely close vents when weighing because disrupting the temperature control system may result in temperature changes near the balances.

Handling Weights

The masses of standard weights or objects weighed can be affected significantly by the manner in which they are handled. Human contact can leave grease or oily films that affect the mass at the time of measurement and can even cause permanent damage due to corrosion.

Small weights should never be touched by hand, but handled using forceps, clean gloves, or swatches of cloth. In the latter case, the cloth must be lint free. Instructions for cleaning weights and for removing adhering foreign material are described in GMP 5.

Large weights of lower tolerance classes (NIST Class F) may be handled by bare hands. Large weights are a source of special problems. Fork lifts, portable cranes, hoists, or even overhead cranes may have to be used to move the weights within the laboratory and on or off the balances. Laboratory personnel must become expert in using such equipment, as necessary, to avoid damage to the laboratory facilities, to the balances used, and even to the weights themselves. Special hoist/crane hydraulics or multi-speed systems are available to gently set large weights in place on large comparators to avoid damage. The problem of temperature equilibrium for large weights is qualitatively the same as for small weights and needs consideration on an individual basis.

Large weights must be clean at the time of use, but cleaning may be a problem. Minimally, they should be inspected to ensure that foreign material is not present. Cleaning is addressed further in GMP 5.

Operation

Analytical balances are high precision instruments and should be operated carefully. Excessive shock can damage a balance. Avoid shock loading the balance. Gently place all weights in the center of the weighing pan/platform. The dials on mechanical balances should be turned slowly and gently. Careful balance operation will improve the repeatability of measurements.

Mechanical analytical balances are provided with partial and full release positions. The partial release position is used when determining if an unknown load will provide an on-scale reading. The balance beam has limited movement in this position. The partial release position provides

some protection to the balance when the dialed-in weights are not close to the actual mass placed on the pan. It is good practice to arrest the pan each time a dial is being changed to protect the balance from shock loading. It is acceptable to change the dial representing the smallest built-in weights when in the partial release position because the small weight changes should not result in shock loading of the balance.

When releasing the pan to either the full or partial release position, the action should be done slowly and carefully. The objective is to minimize disturbances to the balance as much as possible.

Similarly, all loads should be placed on the balance pan carefully and centered on the pan.

When a mechanical balance is released, the beam goes through a series of oscillations. The frequency of these oscillations diminishes as time passes until they are almost imperceptible to the naked eye. At this point, optimal stabilization is achieved. This stabilization of the balance typically lasts for a very short period of time, after which the balance reading will usually drift. A similar situation occurs when a mass is placed on the pan of an electronic balance. Therefore readings should be taken at the precise moment of achieving balance stability. This interval between the releasing of a pan on a mechanical balance, or the placing of a mass on an electronic balance, and the reading of the observation, varies from balance to balance. Stabilization time differs for all balances, even among those of the same type and model. Knowledge of the instrument is critical to correctly establish this time interval. Although manufacturers will usually state this value, it is necessary for the metrologist to verify its reliability. Many electronic balances have a stability signal incorporated into the design, but this also must be verified. All measurements in a calibration should be performed at the same time interval, and within the shortest time possible.

All balances should be exercised before taking readings. A load equal to the load to be measured should be placed on the balance, a reading taken, and the pan arrested, if appropriate, or the weight removed from electronic balances. This operation should be repeated several times before readings are taken for measurement purposes. Once the balance has been "warmed-up", better repeatability will be achieved. Balances can be very accurate even when used without being exercised first, but improved results can be obtained by going through a "warm-up" procedure. If the larger variation present in initial weighings on a balance that has not been exercised is not significant to the measurement, the warm-up procedure may be minimized.

To determine the repeatability of measurements when a balance has not been exercised versus its performance after being exercised, and to determine how much warm-up time is required, begin measurements starting with the unexercised balance and record the readings. Repeat a series of measurements until you have obtained several measurements after the balance has been exercised. This test can be repeated over several days using the same objects and the results compared. The readings obtained when using an unexercised balance are likely to show a slightly larger variation than those obtained after the balance has been exercised. Balance drift is likely to be larger initially and then reach a steady state when the balance has been "warmed-up".

Comparison Weighing - Mechanical Balance

For mass calibrations, the unknown object must be compared to a known mass standard. Comparison weighing eliminates the errors of the built-in weights, reduces disturbances during the measurement because dial settings are not changed during the measurement, and can cancel the effect of drift by selecting the appropriate weighing design. Comparing the unknown, *X*, to a standard, *S*, eliminates the built-in weights from the measurement: thus, the built-in weights act only as counterweights; they do not affect the difference measured between *X* and *S*. Consequently, the dial settings must not be changed during a comparison measurement; otherwise the built-in weights would be part of the measurement.

When comparison measurements are made on a single pan mechanical balance, all readings are taken from the optical scale. The unknown and the standard must have nearly the same mass so that the difference between them can be measured on the optical scale. If the masses of the unknown and the standard are significantly different, small mass standards are used as tare weights with either the unknown or the reference standard or both to obtain an observed mass difference that is significantly less than one-fourth the range of the optical scale.

As part of a comparison measurement, the mass value of a scale division is determined by performing a sensitivity determination. The small mass standard used as part of the weighing design to calibrate the optical scale is called the sensitivity weight and should have a mass between 1/5 and 1/2 the range of the optical scale, inclusive. Additionally, the mass of the sensitivity weight should have a mass that is at least twice the observed mass difference between the standard and the unknown. Since the maximum size of the sensitivity is limited to one-half the range of the optical scale, it may be necessary to carry tare weights with the standard and the unknown to ensure that the observed difference between them is less than one-half the mass of the sensitivity weight. The value of the sensitivity weight should include the correction plus its nominal value. For high precision calibrations, the inclusion of the air buoyancy correction for the sensitivity weight is critical. See GMP 14 for detailed guidance on the selection of sensitivity weights.

The first readings for the standard and the unknown in a comparison on a single pan balance should fall in the first quarter of the optical scale, but well ahead of zero, so the balance drift will not result in negative values for any readings. Although negative numbers may be used in calculations, they are avoided to simplify calculations and reduce calculation errors. Because the sensitivity weight may have a mass as large as one-half the range of the optical scale and the measured difference between the standard and the unknown may be as large as one-fourth the range of the optical scale, it is necessary to obtain the first two readings in the first quarter of the optical scale so all readings will remain on-scale for the measurement. In this way it is not necessary to change the dial settings to measure the difference between the standard and the unknown.

Comparison Weighing - Electronic Balance

Measurements made on a full electronic balance are simplified because there are no built-in weights to consider. Although many electronic balances are equipped with a built-in calibration weight, the weight is not involved in the comparison weighing.

The principles for comparison weighing on a full electronic balance are the same as when using a single pan mechanical balance. The balance indications are used to measure the mass difference between the standard and the unknown, and a sensitivity weight is used to establish the mass value for a digital division on the balance. Since there are no built-in weights in the full electronic balance, the entire range of the digital indications can be considered for "optical scale" of the balance.

For comparison weighing the standard and the unknown should be "nearly the same mass." Since a full electronic balance has a much larger range for indicating mass values, the masses do not have to be as close together as when a mechanical balance is being used. When using an electronic balance, the difference in mass between the standard and unknown should be less than 0.05 % of the balance capacity. Tare weights that are known standards should be used if the masses are not sufficiently close together. The sensitivity weight used to determine the mass per digital division should have a mass that is at least 10 to 20 times the mass difference between the standard and the unknown but not exceeding 1 % of the balance capacity. For high precision weighing, air buoyancy corrections must be made for all objects used in the intercomparison.

Magnetic and Electrostatic Fields

Care must be taken when weighing magnets or objects having electrostatic charges. A magnetic field will likely affect results of a mass measurement. A magnet is attracted to ferromagnetic materials in the balance and balance chamber. The magnetic field may also affect the magnetic field generated in an electronic balance that utilizes the principle of magnetic force restoration as its method of measurement.

Weights made of ferromagnetic material can become magnetized during manufacture or during use if they are placed in a strong magnetic field. Magnetized weights can result in measurement errors that may go undetected. If a measurement problem is suspected, the weights should be checked for magnetism and may have to be rejected if excessively magnetized.

If magnets or magnetized material must be weighed, the material should be placed in a magnetically shielded container to prevent the magnetic field from generating measurement errors. If balance design and conditions permit, an alternative is to position the magnetized material a relatively large distance from the balance pan using a non-ferromagnetic object on the pan to serve as a platform for the magnetic. Since the strength of the magnetic field drops off at a rate of the cube of the distance from the magnetic, it may be possible to effectively isolate the magnet from other ferromagnetic material in the balance.

Electrostatic fields can also cause errors in measurements. If there is a static charge in a mechanical balance, the balance may give erratic readings and lack repeatability. If the object

being weighed has a significant electrostatic charge, it may result in measurement errors and may leave a static charge on the balance. Electrostatic charges are of particular concern when plastic containers are placed on the balance.

Care should be taken to remove electrostatic charges from objects being weighed by grounding the objects, if necessary, before placing them on the balance. To prevent the build-up of static electricity in a balance, the relative humidity in the laboratory should be maintained between 40 % and 60 %. The water vapor in the air will serve to drain off electrostatic charges from the balance.

Balances utilizing the magnetic force restoration principle for weighing should be checked to verify that the magnetic field generated by the magnetic cell in the balance does not exist around the balance pan. If the shielding of the magnetic cell is inadequate, measurement errors may occur when weighing ferromagnetic objects or when the balance is placed on a surface comprised of ferromagnetic material.

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 evidence of metrological traceability.** The following practice establishes calibration intervals for standards and instrumentation used in measurement processes.

Note: GMP 11 provides the basis for documenting calibration intervals. This GMP is a template that must be modified beyond Section 4.1 to match the scope[1] and specific measurement parameters and applications in each laboratory. Legal requirements for calibration intervals may be used to supplement this procedure, but are generally established as a maximum limit assuming no evidence of problematic data.

1.2. Prerequisites

1.2.1. Calibration history and valid calibration certificates for all laboratory standards.

1.2.2. Up-to-date measurement assurance and uncertainty data for all measurement processes in the laboratory that match the laboratory Scope.

1.2.3. Expected tolerance limits where applicable.

1.3. Safety

1.3.1. No outstanding safety concerns

1 The laboratory scope includes the measurement parameters, range of nominal values, associated uncertainties, and calibration and/or test methods.

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 according to this procedure.

2.2. Apparatus

None.

2.3. Procedure

2.3.1. Identification of Parameters

The laboratory must identify all measurement parameters associated with the Scope of calibrations and tests that will be performed in the laboratory. All standards used in a calibration process must be assessed to determine their level of significance.

2.3.2. Standards are identified as being critical parameters or secondary parameters.

2.3.2.1. 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.

2.3.2.2. 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.

2.3.3. Assignment of Initial Intervals

Assignment of initial intervals is based on examples and recommendations given in this GMP. Otherwise, an 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 a control chart when the investigation yields no apparent correctable cause or when results from proficiency tests fail the normalized error assessment (or when the observed bias exceeds the

laboratory uncertainty), or the maximum authorized through law or technical review.

2.3.4. Initial Intervals – Additional Standards

A calibration interval must be documented for all standards used in the laboratory to comply with the definition and interpretation of metrological traceability. Statements such as "as needed" are not acceptable alone without additional qualifications. A statement such as "calibrations are conducted on an annual basis and may be extended based on demonstrated control through use of control charts, surveillance testing, or interlaboratory comparisons, but will not exceed five years" would be acceptable.

2.3.5. Absence of Control Charts or Measurement Assurance Data

If no initial measurement assurance data is available, the laboratory's Technical Manager may assign initial intervals based on evidence observed in the calibration certificate history, engineering evidence, manufacturer′s specifications, NIST recommendations, and valid technical experience.

2.4. Adjustment of Intervals

2.4.1. Intervals will be adjusted when determined to be necessary by the laboratory's Technical Manager. Subsequent intervals may be adjusted based on analysis of measurement assurance data, demonstrated control through surveillance assessments, and ongoing stability as demonstrated through interlaboratory comparisons or proficiency tests.

2.4.2. The intervals may be adjusted by performing a technical analysis, taking the following factors into consideration as appropriate, and documenting the assessment:

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

2.4.3. Inadequate reasons for adjusting intervals might include failure to have an adequate system for tracking due dates, lack of funding for suitable calibrations, or loss of competent staff. Extending calibration intervals must be based on valid, technically supported, and documented assessments.

3. 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 measuring instruments.

4. Calibration Intervals for the Laboratory Scope

4.1. As this document is a template, the laboratory may eliminate all examples in this section and reference another laboratory document that combines metrological traceability information and calibrations in one file as long as it completely states calibration intervals. It is NOT recommended to include this in the laboratory Quality Manual so that updates can regularly be implemented when calibrations are performed.

4.2. Example for Mass

4.2.1. Mass Critical Parameters

4.2.1.1. Balance and Measurement Process Performance

Balance service, maintenance, and calibration must be a regular part of the laboratory operations and is generally performed on a regular periodic basis (often annually) unless problems are observed.

Balance performance in weighing processes following SOP 5 and SOP 28 (e.g., Echelon I) is evaluated in each measurement series. An F-test ratio evaluates the observed standard deviation of the process against the accepted standard deviation of the process.

All other weighing processes have incorporated measurement control procedures and control charts that must be evaluated for balance performance characteristics as data is collected.

Table 1. Balances used for mass (and gravimetric volume) calibrations.

Item	Initial Service and Calibration Interval (months)	Source
Balances and Mass Comparators	12	Approved Provider

4.2.1.2. Mass Standards

Each weighing series in Echelon I also incorporates a check standard with a value that is evaluated using a t-test. The observed value is compared to the accepted reference value to determine if there might be problems. 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 2. Example mass standard calibration intervals where Echelon I is in place.

Item	Initial Cal Interval (months)	Source
R 1. kg and R 1.. kg	48	NIST
S_c 1. kg and S_c 1.. kg	(alternating 2 years) 48	NIST
R 30 kg to R 2 kg	12	Lab
R 500 g to R1 mg	12	Lab
S_c 500 g to to S_c 1 mg	12	Lab
W 25 kg to W 1 mg	12	Lab
R 500 lb to 1 μlb	12	Lab
W 5000 lb to 1 μlb	12	Lab
R = Reference; S_c = check/control; W = working standards		

Note: where Echelon I is not in place, all reference standards or working standards may need to be calibrated by a suitable calibration provider.

4.2.2. Mass Secondary Parameters

Availability of multiple units for environmental equipment enable the laboratory to conduct internal comparisons immediately after a calibration or between calibration cycles and generate internal calibration reports to enable extension of calibration intervals as long as adequate stability is demonstrated. See procedure for adjusting calibration intervals. The following table contains initial calibration intervals.

Table 3. Example calibration intervals for environmental equipment.

Item	Initial Cal Interval (months)	Source
Barometer	12	Accredited Lab
Hygrometer	24	Accredited Lab
Thermometer (digital)	12	Accredited Lab

4.3.Example for Length

4.3.1. Length Critical Parameters

4.3.1.1.Length Standards

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 4. 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

4.3.2. Length Secondary Parameters

Table 5. Calibration intervals for length secondary parameters.

Item	Initial Cal Interval (months)	Source
Thermometer (digital)	12	Accredited Lab

4.4.Example for Volume

4.4.1. *Volume Critical Parameters*

4.4.1.1.Volume Standards

Volume standards are dynamic with use. Wear, contamination and other factors can cause drift from accepted values. Seals and valves must regularly be assessed for leaks and stability. Initial calibration intervals are as follows:

Table 6. Calibration intervals for volume standards.

Item	Initial Cal Interval (months)	Source
R 100 gal standard	60	NIST
R 25 gal standard *	60	Lab
R 5 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.		

4.4.2. Volume Secondary Parameters

Table 7. Calibration intervals for volume secondary parameters (gravimetric).

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

Table 8. Calibration intervals for volume secondary parameters (transfer).

Item	Initial Cal Interval (months)	Source
Thermometer (digital)	12	Accredited Lab

4.5.Example for Thermometry

4.5.1. *Thermometry Critical Parameters*

4.5.1.1.Temperature Standards

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

Table 9. Calibration intervals for temperature standards.

Item	Initial Cal Interval (months)	Source
25.5 ohm SPRT	36	NIST
100 ohm PRT′s	12	Accredited Lab
Standard Thermistor	12	Accredited Lab
Check Standards	12	Accredited Lab
Liquid-in-glass standards	120*	Accredited Lab

**Annual inspection should also ensure that there is no damage or separation in the liquid column.*

Where internal intrinsic or reference standards are available, and the laboratory has the demonstrated competency, the following may be used to set or adjust calibration intervals: triple point cells, melting point cells, and ice baths (using documented procedures).

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GMP 12

Good Measurement Practice
on
Standard Operating Procedure Selection

Good laboratory practices, use of proper standards and equipment, and selection of standard operating procedures are essential for providing calibration results with accurate and traceable values with appropriate and suitable uncertainties. The following matrix recommends SOPs based on the parameter, type of test items, and level of uncertainty needed.

Parameter	Test Item	Recommended SOP
Mass (NISTIR 6969, 2014 and NISTIR 5672 2014)		
	Railroad test cars	SOP 27, Railroad Test Cars using a Master Track Scale
	Weight carts	SOP 33, Calibration of Weight Carts (References SOP 4 and SOP 7)
HB 105-1, Class F ASTM, OIML	Class F Class 5, 6, 7 Class M1, M2, M3 e.g., 10 kg to 250 kg (≥500 lb) cast iron 1 mg to 5 kg (1 µlb to 10 lb) stainless steel	SOP 8, Modified Substitution - may be used if expanded uncertainty is less than 1/3 of the tolerance SOP 7, Single Substitution - to be used, as a minimum, if conditions given for SOP 8 cannot be met NOTE: Balances and standards must be selected properly for these conditions to be met.
ASTM, OIML	Class 3, 4 (P) Class F1, F2 e.g., 1 kg kit, 100 g kit	SOP 7, Single Substitution - may be used if expanded uncertainty is less than 1/3 of the tolerance. If uncertainty is greater than 1/3 of the tolerance, then use SOP 4. SOP 4, Double Substitution - to be used for buoyancy corrections and if expanded uncertainty is less than 1/3 of the tolerance Modified SOP 4/5, to be used to incorporate measurement control into SOP 4 NOTE: Balances and standards must be selected properly for these conditions to be met.
ASTM, OIML	Class 1, 2 (S, S-1) Class E2 for use in balance calibration	SOP 5, 3-1 Weighing Design (preferred) Modified SOP 4/5, to be used to incorporate measurement control with SOP 4

Parameter	Test Item	Recommended SOP
ASTM, OIML	Class 0, 1 (S) Class E1 for use as laboratory standards	Weighing Designs per TN 952, TN 844, NISTIR 5672, SOP 28, with appropriate check standards; SOP 5 may be used as well.
Volume (NISTIR 7383, 2013)		
HB 105-2	Glass flasks	SOP 14, Gravimetric Calibration of Volumetric Standards Using an Electronic Balance SOP 16, Calibration of Measuring Flasks Volume Transfer Method (acceptable)
HB 105-3	20 L test measures (5 gal or 10 gal)	SOP 19, Calibration of Graduated Neck-Type Metal Provers (Volume Transfer Method) is preferred. When temperature instability is observed during the calibration process, SOP 19 must be used. SOP 18, Calibration of Graduated Neck-Type Metal Volumetric Field Standards Using a Slicker-Plate Type Standard, may be used if temperature stability and limits are acceptable (single delivery from slicker plate type standard; if glass standards use SOP 19.
HB 105-3	Large graduated neck type provers - used for meter verification	SOP 19, Calibration of Graduated Neck-Type Metal Provers (Volume Transfer Method)
HB 105-4	LPG provers	SOP 21, Calibration of LPG Provers
HB 105-7	Dynamic Small Volume Provers	SOP 26, Gravimetric Calibration of Dynamic Volumetric Systems used as Standards
	Laboratory standards Glassware: burets, pipetes, flasks	SOP 14, Gravimetric Calibration of Volumetric Standards Using an Electronic Balance
	Laboratory standards Laboratory slicker plate standards	SOP 14, Gravimetric Calibration of Volumetric Standards Using an Electronic Balance
	Micropipetes	SOP 14, Gravimetric Calibration of Volumetric Standards Using an Electronic Balance

Parameter	Test Item	Recommended SOP
HB 105-3	Large graduated neck type provers - used as laboratory standards	SOP 19, Calibration of Graduated Neck-Type Metal Provers (Volume Transfer Method) OR SOP 14, Gravimetric Calibration of Volumetric Standards Using an Electronic Balance
Length		
	Tapes	SOP 11, Bench Method (lower uncertainties) OR SOP 12, Tape to Tape
	Rigid Rules	SOP 10, Rigid Rule
	Pi Tapes	SOP 23, Pi Tape Calibration
Liquid-in-Glass Thermometers		
HB 105-6	Field standards for weights and measures	SOP 25 (unpublished)
Timing Devices		
HB 105-5	Field standards for weights and measures	SOP 24
Traffic Speed Gun Tuning Forks		
	For highway official use	SOP 22 (unpublished)

Parameter	Process	Recommended SOP
Measurement Assurance		
All Process Measurement Assurance	SOP 30	Use of process measurement assurance programs
Mass	SOP 5, 28	Use of check standards in procedure TN 952, TN 844, NISTIR 5672
Mass	SOP 4, 6, 7, 8	SOP 9
Length	SOP 10, 11, 12, 23	Redundancy built into procedures with replicate measurements

Parameter	Process	Recommended SOP
Measurement Assurance		
Volume	SOP 13, 14, 15, 16, 18, 19, 21, 26	SOP 17, laboratory check standards OR SOP 20, standard deviation charts and range charts
Temperature	SOP 25	Use of check standards in procedure
Uncertainty		
All parameters	All SOPs	SOP 29, Calculation of Uncertainty

GMP 13

Good Measurement Practice for Ensuring Metrological Traceability[1]

1 Introduction

1.1 Purpose

The purpose of this Good Measurement Practice is to enable compliance with essential elements of Metrological Traceability. Traceability ensures that the measurements are accurate representations of the specific quantity subject to measurement, within the uncertainty of the measurement.

To ensure metrological traceability, suitably calibrated standards that are appropriately maintained and cared for, proper standard operating procedures, continuous measurement control, surveillance, and suitable documentation must all be present.

Test numbers issued by NIST should not be used nor required as proof of the adequacy or traceability of a test or measurement. Having a NIST number does not provide evidence that the measurement value provided by another organization has the property of metrological traceability.

GMP 13 provides the basis for documenting metrological traceability. This GMP is a template that must be modified beyond Section 4 to match the laboratory scope, specific measurement parameters, and uncertainties in each laboratory.

1.1.1 **Metrological Traceability** is defined[2] as the "property of a measurement result whereby the result can be related to a reference through a documented unbroken chain of calibrations, each contributing to the measurement uncertainty."

NOTE 1: For this definition, a 'reference' can be a definition of a measurement unit through its practical realization, or a measurement procedure including the measurement unit for a non-ordinal quantity, or a measurement standard.

NOTE 2: Metrological traceability requires an established calibration hierarchy.

NOTE 3: Specification of the reference must include the time at which this reference was used in establishing the calibration hierarchy, along with any other relevant

1 See also the International Laboratory Accreditation Cooperation (ILAC) Policy ILAC P10:01/2013, ILAC Policy on the Traceability of Measurement Results. Available http://ilac.org/publications-and-resources/ilac-documents/procedural-series/ (August 2014).
2 These definitions are provided in the "International vocabulary of metrology — Basic and General Concepts and Associated Terms (VIM)" JCGM 100:2008(2012).

metrological information about the reference, such as when the first calibration in the calibration hierarchy was performed.

NOTE 4: For measurements with more than one input quantity in the measurement model, each of the input quantity values should itself be metrologically traceable and the calibration hierarchy involved may form a branched structure or a network. The effort involved in establishing metrological traceability for each input quantity value should be commensurate with its relative contribution to the measurement result.

NOTE 5: Metrological traceability of a measurement result does not ensure that the measurement uncertainty is adequate for a given purpose or that there is an absence of mistakes.

NOTE 6: A comparison between two measurement standards may be viewed as a calibration if the comparison is used to check and, if necessary, correct the quantity value and measurement uncertainty attributed to one of the measurement standards.

NOTE 7: The ILAC considers the elements for confirming metrological traceability to be an unbroken metrological traceability chain to an international measurement standard or a national measurement standard, a documented measurement uncertainty, a documented measurement procedure, accredited technical competence, metrological traceability to the SI, and calibration intervals (see ILAC P-10:2002).

NOTE 8: The abbreviated term "traceability" is sometimes used to mean 'metrological traceability' as well as other concepts, such as 'sample traceability' or 'document traceability' or 'instrument traceability' or 'material traceability', where the history ("trace") of an item is meant. Therefore, the full term of "metrological traceability" is preferred if there is any risk of confusion.

1.1.2 A **Metrological Traceability Chain** is defined as "traceability chain sequence of measurement standards and calibrations that is used to relate a measurement result to a reference".

NOTE 1: A metrological traceability chain is defined through a calibration hierarchy.

NOTE 2: A metrological traceability chain is used to establish metrological traceability of a measurement result.

NOTE 3: A comparison between two measurement standards may be viewed as a calibration if the comparison is used to check and, if necessary, correct the quantity value and measurement unit.

1.1.3 **Metrological Traceability to a Measurement Unit** is defined as: metrological traceability to a unit metrological traceability where the reference is the definition of a measurement unit through its practical realization

NOTE: The expression "traceability to the SI" means 'metrological traceability to a measurement unit of the International System of Units'. (See section 1.3.3.)

1.2 Prerequisites

Metrological traceability can be characterized by the following seven essential elements:

1.2.1 *Unbroken chain of comparisons.* A documented system of comparisons with each step having the essential elements of metrological traceability going back to a standard acceptable to the parties, usually a national or international standard;

1.2.2 *Documented Measurement Uncertainty.* The measurement uncertainty for each step in the traceability chain must be calculated according to defined methods and must be stated so that an overall uncertainty for the whole chain may be calculated (see SOP 29);

1.2.3 *Documented Measurement Procedure.* Each step in the chain must be performed according to documented and generally acknowledged procedures (see GMP 12) and the results must be documented (i.e., in a calibration certificate, see SOP 1);

1.2.4 *Accredited Technical Competence.* The laboratories or bodies performing one or more steps in the chain must supply evidence of technical competence (e.g., by maintaining appropriate training records, participating in interlaboratory comparisons, and by demonstrating that they are accredited by a recognized accreditation body);

1.2.5 *Realization of SI Units.* The primary national, international or intrinsic standards must be primary standards for the realization of the International System of Units (SI);

1.2.6 *Documented Calibration Intervals.* Calibrations of standards (and equipment where appropriate) must be repeated at established (may be defined through measurement assurance) and appropriate intervals to preserve metrological traceability of the standard over time and use (see GLP 4, GMP 11); and

1.2.7 *Measurement assurance.* A proper measurement assurance program must be established to ensure the validity of the measurement process and the accuracy of standard used at the time of the measurement (see SOPs 9, 17, 20, 30).

1.3 Responsibility

1.3.1 *Provider.* Providing support for the claim of traceability of the result of a measurement or value of a standard is the responsibility of the calibration provider. Calibration certificates must contain a statement regarding metrological traceability. See Appendices C and D for a form that may be used to assess evidence supporting metrological traceability.

1.3.2 *User.* Assessing the validity of a claim of traceability is the responsibility of the user of that result or value. Verifying claims of traceability often includes obtaining a calibration directly from a national metrology institute or another laboratory that has achieved recognition or accreditation through a recognized accrediting body. See Appendix E for a form that may be used to assess calibration certificates and standards for weights and measures applications.

1.3.3 *Use of, or reference to, official test numbers of a national metrology institute.* Having an authentic test number does not provide assurance or evidence that the measurement value provided by another organization is traceable. Not only must there be an unbroken chain of comparisons, but each measurement must be accompanied by a statement of uncertainty associated with the value. Test report numbers should not be used nor required as proof of the adequacy or traceability of a test or measurement. National and international standards dealing with test and measurement quality requirements, such as ISO 10012, ISO/IEC 17025 and the ISO 9000 series do not require the use or reporting of specific test numbers to establish traceability.

1.4 Safety

No outstanding safety concerns

2 Methodology

2.1 Summary

Traceability must be maintained through comparison to appropriate standards with suitable procedures and measurement uncertainties. Procedures are outlined in SOPs and GMPs. Examples of possible hierarchies of the standards leading to the traceability of a calibration are provided in this document in Appendix A.

2.2 Procedure

2.2.1 Create Traceability Hierarchy Charts for the Laboratory

The charts in this GMP show examples of the traceability hierarchy for mass, length, volume, and temperature measurement disciplines. Each laboratory must define the exact traceability system in their operating quality manual and have evidence of all essential elements of traceability confirmed to perform associated calibrations. (A

worksheet is included as Appendix B to help in the definition of the traceability system. The worksheet may be used as a template and combined with requirements for establishing calibration intervals and identifying calibration sources.)

2.2.2 Tools for assessing the evidence of metrological traceability

- Appendix C – Traceability Evaluation Form – for assessing laboratory evidence of traceability
- Appendix D – Sample Technical Audit for Traceability Evidence – tool for selecting calibration certificates and assessing the evidence in the laboratory as part of a technical audit; and
- Appendix D – Evaluating Supplier Calibration Certificates for Metrological Traceability for Weights and Measures Applications – tool to assess traceability for weights and measures applications as needed

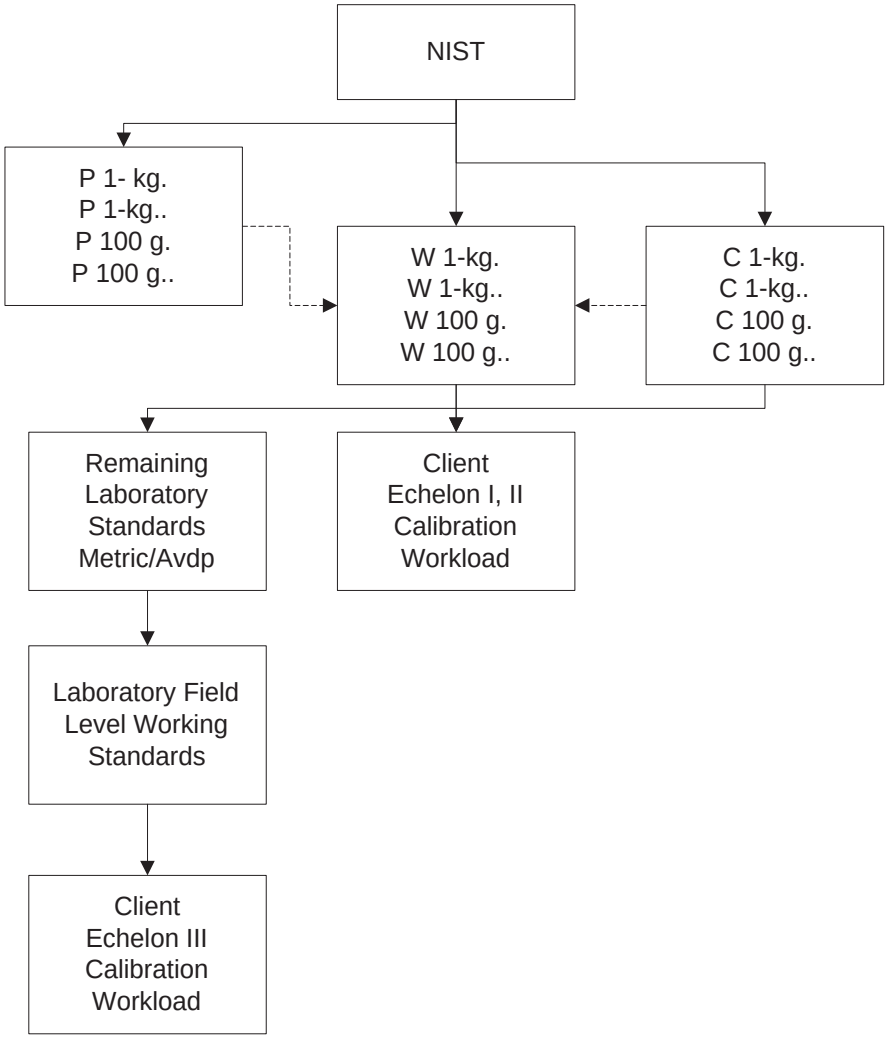
3. Calculations

There are no calculations in this GMP.

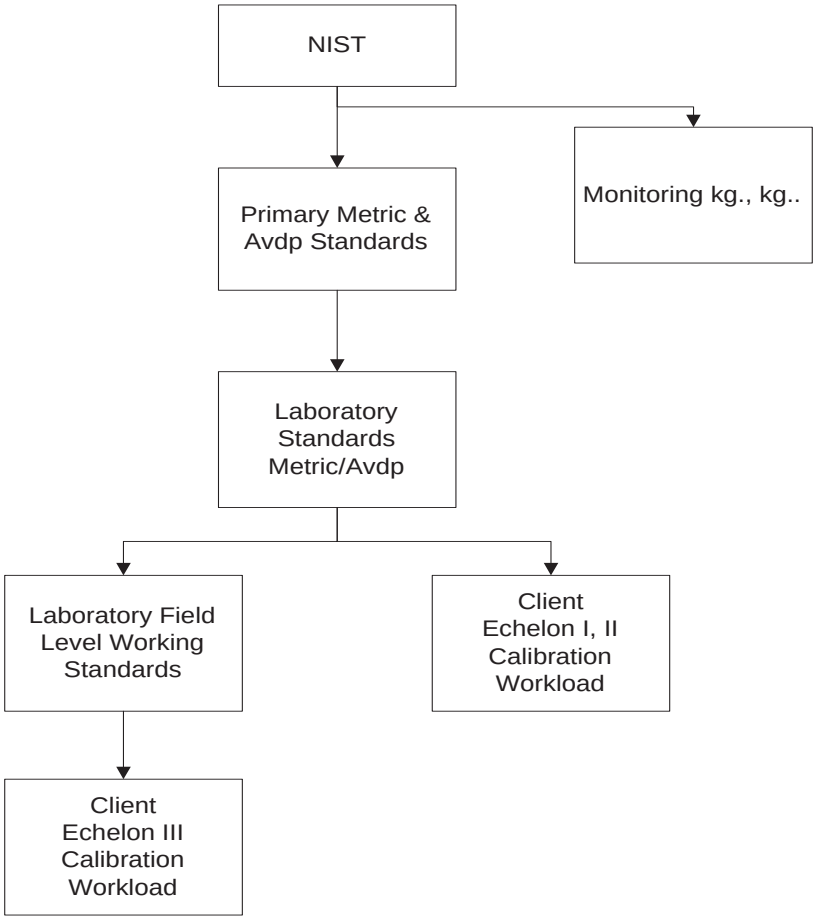
4. Assignment of Uncertainty

The uncertainty associated with reported calibration values is included within the uncertainty analysis for each SOP and in SOP 29, Calculating and Reporting Uncertainties.

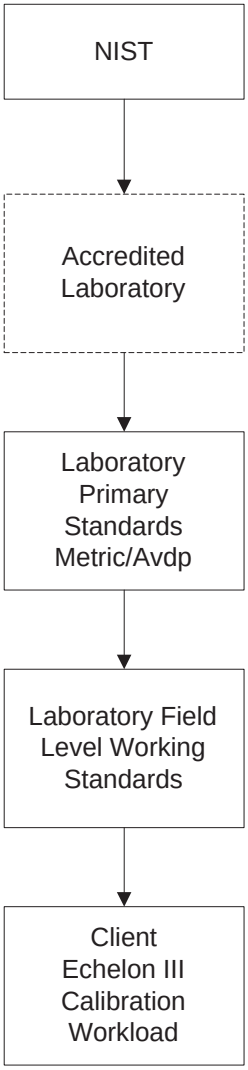
Appendix A – Examples
Mass - Example A



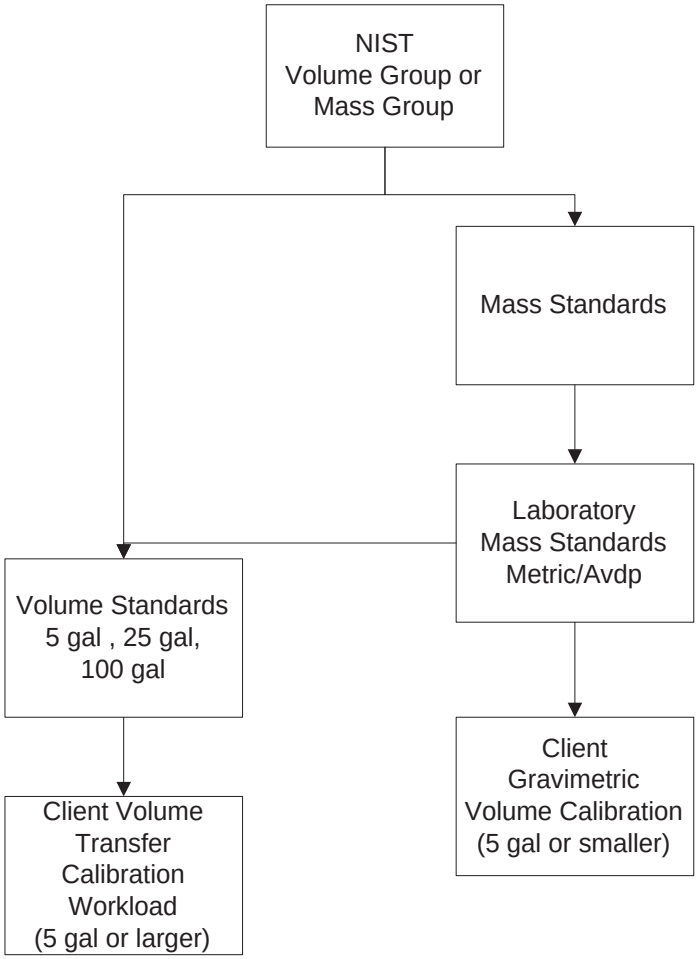
Mass - Example B



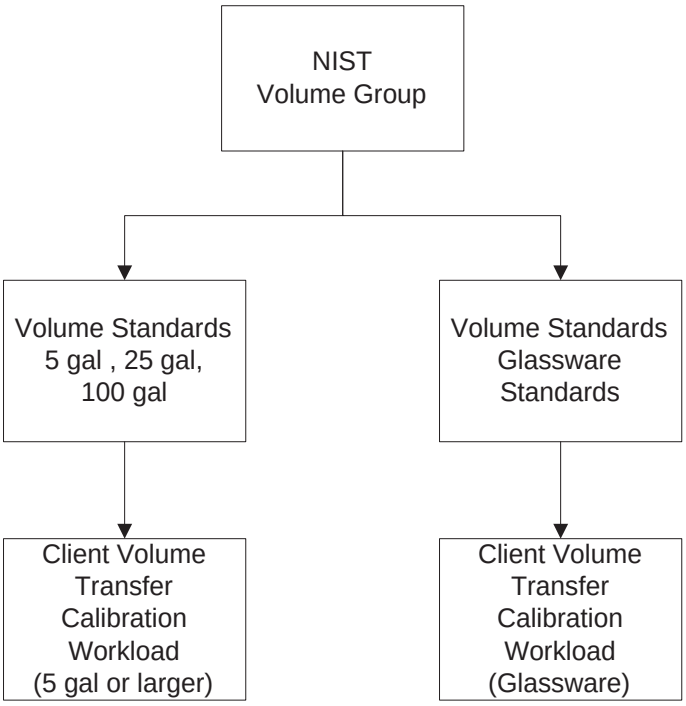
Mass - Example C



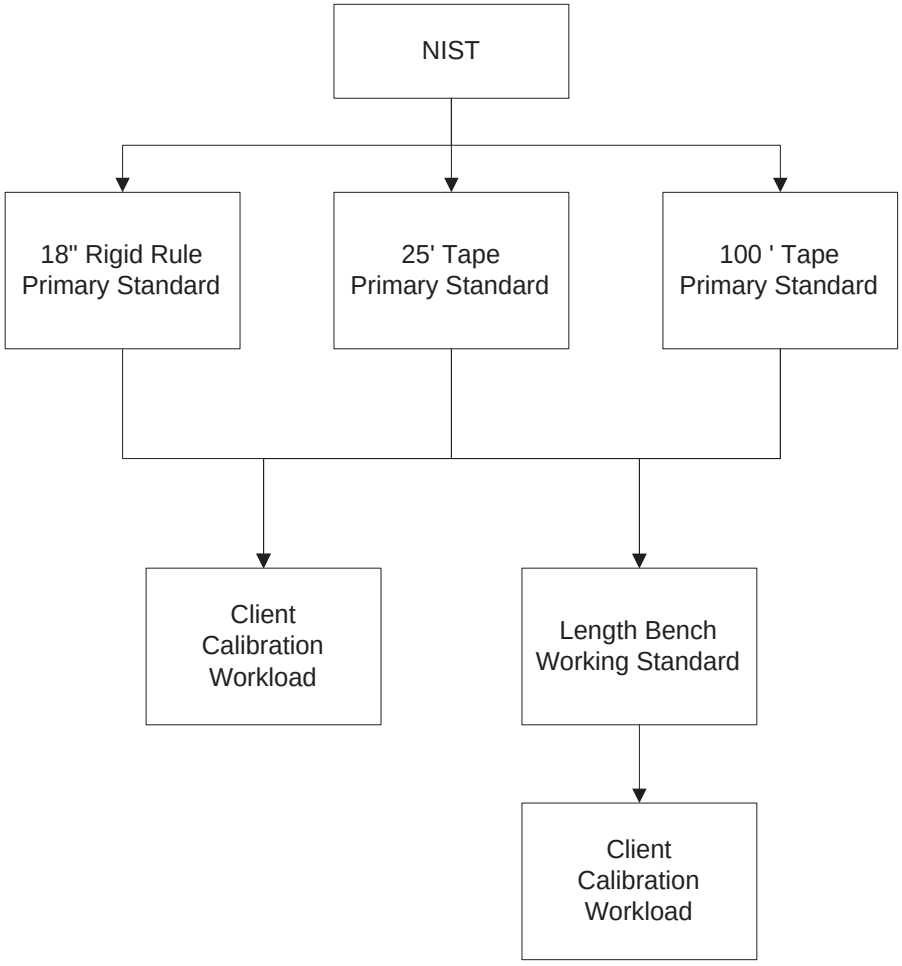
Volume - Example A



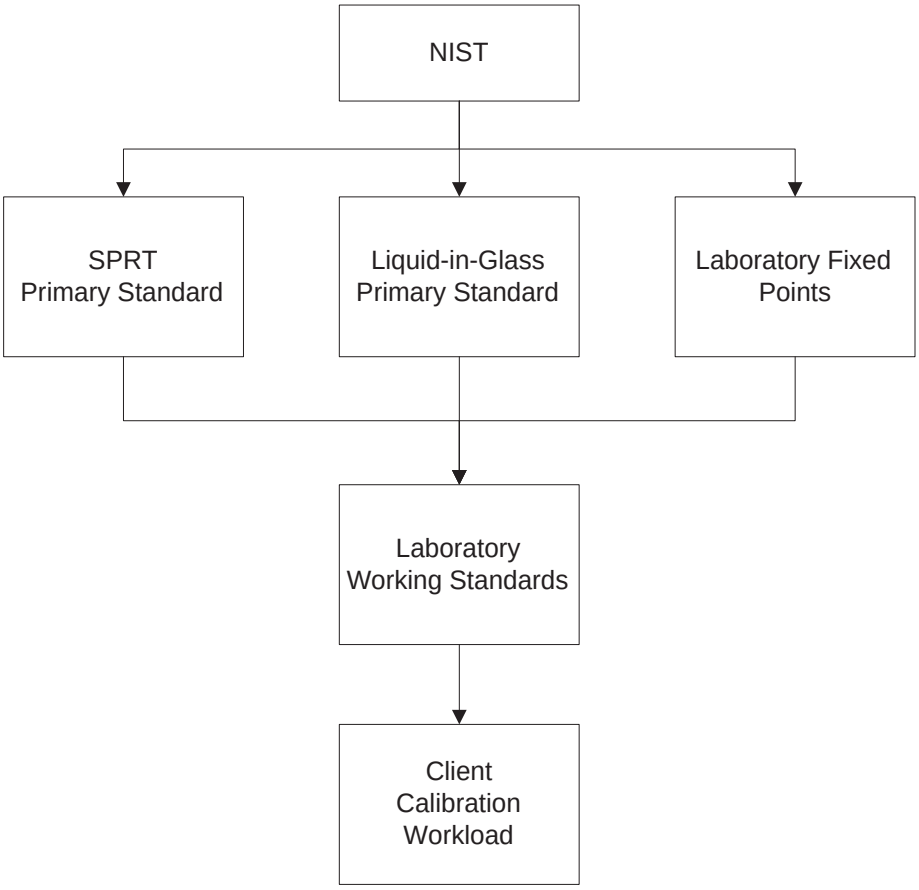
Volume - Example B



Length Example



Temperature Example



Appendix B - Traceability worksheet

Parameter: Mass, Volume, Length, Temperature, Other

R		SI (g, L, m, °C)	Cal. date	Source	Interval
	Range				
		Customary (lb, gal, ft, °F)			
	Range				

W		SI (g, L, m, °C)	Cal. date	Source	Interval
	Range				
		Customary (lb, gal, ft, °F)			
	Range				

S_c		SI (g, L, m, °C)	Cal. date	Source	Interval
	Range				
		Customary (lb, gal, ft, °F)			
	Range				

R = Reference Standards; W = Working Standards; S_c = Check Standards.

Appendix C: Traceability Evaluation Form

Assess whether all of the essential elements of traceability are available for the entire Scope by completing the table below. Specific Analysis: Include specific comments on what was observed and what evidence is available.

Manual Reference	Description	Specific Example or Evidence	Complies? Yes/No
	Unbroken Chain of Evidence. Standards (Reference, Working, and Check or Control) demonstrate an unbroken chain to SI references.		
	Documented Uncertainty. Needed for each step in the hierarchy.		
	Documented Procedures. Suitable, applicable, documented and validated procedures.		
	Accredited Technical Competency. Demonstration of competency is documented.		
	Realization to SI Units.		
	Calibration intervals.		
	No calibration intervals are "past due."		
	Measurement Assurance.		

Appendix D: Sample Technical Audit for Traceability Evidence

Select 3 calibration certificates for the past year, and identify the following specific information to support traceability for each calibration. On each chart, next to each block for your primary and working standards, please list evidence that is available in your laboratory:

Item Evaluated	Evidence
Calibration selected (Measurement Type, Lab Test Number)	
Test Number(s) for all standards used	
Calibration Date(s) for the standards	
Calibration Interval	
Uncertainty analysis components included(s)	
Uncertainty file name where data is stored	
Documented SOP used	
Evidence of competency	
Measurement assurance type	
Measurement Assurance file name where data is stored	
Report contains a Traceability Statement	

You should be able to identify any gaps and corrective action needed in your laboratory in the area of traceability, uncertainty analysis, and measurement assurance. Note it below.

Appendix E: Evaluating Supplier Calibration Certificates for Metrological Traceability for Weights and Measures Applications

Step	Notes	Accept (Yes/No)
Legal	Does your law allow acceptance of reports for the applicable use (laboratory standards, field standards, service company standards)? Does your law specify traceability to NIST? To the SI? Is there a required calibration interval?	
Specifications	Does your law require assessment against published specifications like the NIST Handbook 105-series documentary standards? IF SO, have the customer standards been evaluated for compliance to specifications (are the standards suitable for use?) Note: This is NOT common for non-governmental laboratories, even when accredited, and cannot be done if there are no applicable specifications. *Accreditation has nothing to do with evaluating compliance to specifications.*	
Documented Calibration Certificate	Is there a valid calibration certificate for *all of* the standards? Does it (do they) comply with laboratory requirements? (Checklist available.) Specifically, traceability components: reference to SI and standards used, traceability statement, uncertainty statement, measurement results, measurement uncertainty, and reference to documented procedures.	
Is the report from a National Metrology Laboratory (NMI) like NIST?	Does your law permit you to accept a calibration certificate for a standard that was tested by Measurement Canada that is traceable to NRC for example? Has that NMI signed the CIPM MRA and is this range/scope/uncertainty of measurements published in the Appendix C listing maintained by BIPM? (List available.)	
Is the report from a NIST OWM Recognized laboratory?	OWM is NOT a recognized Accreditation Body and has not signed the ILAC MRA. Is the measurement parameter on their Scope? Is the reported uncertainty acceptable for your needs? (Posted on our website, includes those Accredited by NVLAP.)	
Is the report from an Accredited laboratory?	Is the Accreditation Body a signatory of the ILAC MRA? (List available.) Is the measurement parameter on their Scope *now*? Is the *currently* reported uncertainty small enough to meet requirements for the laboratory or for Fundamental Considerations (less than 1/3 the applicable tolerance)?? (Directories available.)	

Standard Operating Procedures

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SOP 1

Recommended Standard Operating Procedure for Preparation of Calibration Certificates

1. Introduction

   1.1. Calibration certificates are the visible outputs of the testing laboratory. They should be prepared with utmost care to ensure that they accurately convey all information pertaining to the testing so that reports may be used with maximum benefit by all concerned. Carefully prepared calibration certificates must contain or refer to all information necessary to justify the test results.

   1.2. The calibration certificate may consist of filling in the blanks in a form in the case of a routine measurement. A more detailed report, including narrative information, may be required for special calibrations or tests.

   1.3. Regardless of the final form, the calibration certificate must contain the basic information described in the following sections (see also Section 5.10 of ISO/IEC 17025[1] and the attached Checklist for review of template reports and those provided by calibration suppliers).

2. Content

   2.1. Title (e.g., "Calibration Certificate").

   2.2. Name and address of the laboratory, or location at which tests were performed.

   2.3. Unique identification of the calibration certificate, and on each page an identification in order to ensure that the page is recognized as part of the calibration certificate, and a clear identification of the end of the certificate.

   2.4. Name and address of the client.

   2.5. Method used – Describe how calibration was performed by reference to SOP(s). In the absence of SOP's, brief but informative descriptions of the methodology should be included. Information describing deviations from previously agreed upon procedure must also be included.

   2.6. Description of, the condition of, and unambiguous identification of the item calibrated (e.g., description and/or serial numbers). A laboratory number should be assigned and attached to each test item at the time of its acceptance for testing. The use of the

[1] ISO/IEC 17025:2005 (reviewed 2010) - General Requirements for the Competence of Testing and Calibration Laboratories.

laboratory number will facilitate the internal control of test items during the testing process.

2.7. Date of receipt of calibration item where this is critical to the validity and application of the results, and the date of performance of calibration.

2.8. Reference to sampling plan or procedures, where relevant.

2.9. Calibration results with the units of measurement in tabular or other convenient form. (When an instrument or standard has been repaired or adjusted the calibration results before and after repair or adjustment are reported.)

2.10. Name, title, and signature of person authoring the report or certificate. Other signatures may be required, at the discretion of the laboratory director. Each signer accepts his/her share of responsibility for the technical accuracy of the report contents.

2.11. Where relevant, a statement to the effect that the results relate only to the items tested or calibrated.

2.12. Conditions (e.g., environmental) under which the calibrations were made that have an influence on the measurement results.

2.13. A statement of the estimated measurement uncertainty, components that were considered and included, a rationale for their inclusion, and the coverage factor and estimated confidence interval. When a standard coverage factor is not consistent for all items that were calibrated, an additional column may be added to the measurement result table.

2.14. Include evidence to support metrological traceability and a traceability statement. E.g., identification of the standards used and their traceability to national or international standards and to the International System of Units (SI), through a national metrology institute as appropriate or as requested by the customer.

2.15. Where relevant, a statement of compliance/non-compliance with requirements and/or specifications. Compliance refers to *all* criteria of a referenced standard and not just a portion (e.g., compliance to all specifications and tolerances versus tolerance only). When all criteria have not been assessed, the laboratory must identify which criteria have/have not been evaluated.

2.16. Where appropriate and needed, opinions and interpretations. When included, opinions and interpretations are clearly stated.

2.17. Additional information which may be required by specific methods, clients or groups of clients.

2.18. Hard copies of calibration certificates should also include the page number and total number of pages.

2.19. A statement specifying that the calibration certificate shall not be reproduced except in full, without written approval of the laboratory.

2.20. Calibration intervals only when required for legal applications or where requested by the customer.

3. Records

3.1. File all calibration certificates in a systematic manner for ease of retrieval, as necessary.

3.2. Retain copies of all calibration certificates according to the laboratory Quality Management System.

3.3. Appendix B may be used as a technical auditing tool to assess the calibration certificate templates of the laboratory and to assess calibration certificates that are submitted by approved suppliers.

APPENDIX A - Example

BUREAU OF STANDARDS
PO Box 12345
City, State 12345-1234

COMPLIANT CALIBRATION LABORATORY
123 Some Ave.
City, State 12312-1231

CALIBRATION CERTIFICATE

FOR

1 kg to 10 mg weight kit

(Twenty-one metric weights)

Maker: DENTROM LAKE
Serial No.: 27269

Lab Test No.: TI-01-056
NMI Test No.: 822/1234

SUBMITTED BY

YOUR CUSTOMER, INC.
Customer's Address
City, State

Nominal (g)	Conventional Mass (g)	Conventional Mass Correction (mg)	Expanded Uncertainty (approximately 95 % confidence interval) (mg)
1 000	1 000.000 82	0.82	0.92
500	500.000 71	0.71	0.53
300	299.999 87	- 0.13	0.27
200	200.000 67	0.67	0.18
100	100.000 411	0.411	0.091
50	50.000 318	0.318	0.051
30	30.000 117	0.117	0.028
20	19.999 987	- 0.013	0.023
10	10.000 011	0.011	0.018
5	5.000 022	0.022	0.015
3	3.000 112	0.112	0.013
2	1.999 965	- 0.035	0.012
1	1.000 117	0.117	0.010
0.500	0.500 013 2	0.013 2	0.005 1
0.300	0.300 022 3	0.022 3	0.004 8
0.200	0.200 001 7	0.001 7	0.004 3
0.100	0.100 001 3	0.001 3	0.004 2
0.050	0.050 001 8	0.001 8	0.004 0
0.030	0.030 001 1	0.001 1	0.003 7
0.020	0.020 000 9	0.000 9	0.003 3
0.010	0.009 999 7	- 0.000 3	0.003 1

The data in the above table of this report only applies to those items specifically listed on this report.

Uncertainty statement:
The combined standard uncertainty includes the standard uncertainty reported for the standard, the standard uncertainty for the measurement process, the standard uncertainty for any uncorrected errors associated with buoyancy corrections, and a component of uncertainty to account for any observed deviations from NIST values that are less than surveillance limits. The combined standard uncertainty is multiplied by a coverage factor (*k*) of 2 to provide an expanded uncertainty, which defines an interval having a level of confidence of approximately 95 percent. The expanded uncertainty presented in this report is consistent with the ISO/IEC Guide to the Expression of Uncertainty in Measurement (2008). The expanded uncertainty is not to be confused with a tolerance limit for the user during application.

Traceability statement:
The Standards of the Compliant Calibration Laboratory are traceable to the International System of Units (SI) through the National Institute of Standards and Technology, and are part of a comprehensive measurement assurance program for ensuring continued accuracy and measurement traceability within the level of uncertainty reported by this laboratory. The laboratory test number identified above is the unique report number to be used in referencing measurement traceability for artifacts identified in this report only.

Supplemental Information

Description of artifacts submitted for testing:

Twenty one metric weights from 1 kg to 10 mg, marked ASTM Class 4. Weights from 1 kg to 1 g: two-piece weights, with assumed density of 8.0 g/cm^3. Weights from 500 mg to 50 mg: sheet weights, with assumed density of 16.6 g/cm^3. Weights from 30 mg to 10 mg: sheet weights, with assumed density of 2.7 g/cm^3.

Conditions of artifacts submitted for testing:

Artifacts showed evidence of improper handling. Fingerprints and dents were visible on the surface of the weights.

Treatment of artifacts prior to testing:

Artifacts were cleaned with cheesecloth and ethyl alcohol. Thermal equilibrium time/conditions: ten days next to balances in mass lab.

Equipment & Standards used for this calibration:

Balance	Range	Standards Used	Calibration due
AT1005	1 kg to 200 g	Set H	2/31/2002
AT106	100 g to 10 g	Set H	2/31/2002
UMT5/6	5 g to 10 mg	Set H	2/31/2002

Assumed Density of Reference Standards:

1 kg to 1 g: 7.94 g/cm^3 500 mg to 10 mg: 8.41 g/cm^3

Procedure used:

Double Substitution (NISTIR 6969, SOP 4, 2014)

Environmental conditions at time of test:

Temperature: 20.1 °C to 20.2 °C Barometric Pressure: 752.7 mm Hg Relative Humidity: 43.35 % to 43.40 %

Date artifacts were received: February 15, 2014 Date of report preparation: March 3, 2014

Date of test: February 25, 2014
Due date per customer's request: February 25, 2014

Josh Balani II

Test performed by: Josh Balani II
Metrology Expert

Member:
NCSLI
NCWM
ASQ

This document does not represent or imply endorsement by the Compliant Calibration Laboratory, NMI, or any agency of the State and/or national governments. This document may not be reproduced, except in full, without the written permission of the Compliant Calibration Laboratory.

Appendix B - Calibration Certificate and Test Report Review Checklist

ISO/IEC 17025:2005 (NIST HB 143:2007), Section 5.10 - Reporting the Results

Criteria Guide		Compliance
5.10.1	Reported accurately, clearly, unambiguously and objectively; in accordance with method instructions; include all info requested by customer, necessary for interpretation of the results, & required by the method	
Test Reports and Calibration Certificates Include at least the following information, unless valid reasons for not		
5.10.2 a)	Title	
5.10.2 b)	Laboratory name & address; location where tests/calibrations carried out, if different from lab	
5.10.2 c)	Unique certificate/test report ID (e.g., SN #); ID on each page; clearly ID document end Note: Hard copies should include page number and total number of pages	
5.10.2 d)	Customer name & address	
5.10.2 e)	ID method used	
5.10.2 f)	Description of, the condition of, & unambiguous identification of the item(s) calibrated/tested	
5.10.2 g)	Item(s) receipt date where critical to validity/application of results; date(s) of cal/test performance	
5.10.2 h)	Reference to sampling plan & procedures used where relevant to validity or application of results	
5.10.2 i)	Calibration/test results with units of measurement, where appropriate	
5.10.2 j)	Name(s), function(s) & signature(s) or equivalent ID of person(s) authorizing the certificate/report	
5.10.2 k)	Where relevant, a statement to the effect that the results relate only to the items tested or calibrated Note: Recommend statement shall not be reproduced except in full, without written lab approval	
Test Reports In addition to 5.10.2, test reports shall include the following		
5.10.3.1a)	Deviations, additions, exclusions from method; info on specific test conditions (e.g., environmental conditions)	
5.10.3.1b)	Statement of compliance/non-compliance with requirements and/or specifications, where relevant	
5.10.3.1c)	Statement on estimated uncertainty, where applicable; uncertainty info needed when: relevant to results validity/application, customer's instructions requires, or uncertainty affects specification limit compliance	
5.10.3.1d)	Opinions and interpretations, where appropriate and needed	
5.10.3.1e)	Additional information which may be required by specific methods, customers or groups of customers	
Test Reports Containing the results of sampling		
5.10.3.2 a-f)	Sampling date, ID substance, material or product, location, diagrams, sketches, photographs; reference plan & procedures; environmental conditions; method/procedure, deviations, additions, or exclusions from specification	
Calibration Certificates In addition to 5.10.2, where necessary		
5.10.4.1 a)	Calibration conditions (e.g., environmental) that have an influence on the measurement results	
5.10.4.1 b)	Uncertainty and/or statement of compliance with an identified metrological specification or its clauses	
5.10.4.1 c)	Evidence that the measurements are traceable	
5.10.4.2	Relate only to quantities/ results of functional tests. IF compliance statement is made: ID clauses met/not met & account for uncertainty; Record & maintain results for reference if results/uncertainties omitted	
5.10.4.3	Report before and after adjustment or repair results, if available, when adjusted/repair are made	
5.10.4.4	No calibration interval on certificate/label except if agreed w/customer (superseded by legal regulations)	
Opinions and Interpretations		
5.10.5	Document basis of opinions and interpretations when included; clearly mark them	
Format of Reports and Certificates		
5.10.8	The format shall be designed to accommodate each type of test or calibration carried out and to minimize the possibility of misunderstanding or misuse	
Amendments to Test Reports and Calibration Certificates		
5.10.9	Amendments after issue made as further document/data transfer with statement, meet all requirements; New report/certificate is uniquely identified & reference to the original that it replaces	

SOP 2

Recommended Standard Operating Procedure for Applying Air Buoyancy Corrections

1. Introduction

1.1. Purpose

If uncorrected, the effect of air buoyancy on the objects being compared is frequently the largest source of error in mass measurement. This SOP provides the equations to be used to correct for the buoyant effect of air. The significance of the air buoyancy correction depends upon the accuracy required for the measurement, the magnitude of the air buoyancy correction relative to other sources of error in the overall measurement process, and the precision of the mass measurement. An air buoyancy correction should be made in all high accuracy mass determinations. The gravimetric volume procedure uses a high accuracy mass determination with the corresponding buoyancy corrections. The Appendix to this SOP provides a brief theoretical discussion of this subject.

1.2. Prerequisites

1.2.1. Verify that (true) mass values or corrections are available for the standards used in the measurement process and that they have demonstrated metrological traceability to the international system of units (SI), which may be to the SI through a National Metrology Institute such as NIST.

1.2.2. Verify that the thermometer, barometer, and hygrometer used have been calibrated, and that they have demonstrated metrological traceability to the international system of units (SI), which may be to the SI through a National Metrology Institute such as NIST, and are in good operating condition as verified by periodic tests or cross-checks with other standards.

2. Methodology

2.1. Scope, Precision, Accuracy

This procedure is applicable to all weighings using a comparison of mass standards. The precision will depend upon the accuracy of the thermometer, barometer, and hygrometer used to determine the air density. When the calculations for the air density and air buoyancy corrections are made, a sufficient number of decimal places must be carried so the error due to the rounding of numbers is negligible relative to the error in the measurement process. Typically, carrying six to eight decimal places is sufficient, but depends upon the precision of the measurement.

2.2. Apparatus/Equipment Required[1]

2.2.1. Calibrated barometer with sufficiently small resolution, stability, and uncertainty (See SOP 2, e.g., accurate to ± 66.5 Pa (0.5 mm Hg)) to determine barometric pressure.

2.2.2. Calibrated thermometer with sufficiently small resolution, stability, and uncertainty (see SOP 2, e.g., accurate to ± 0.10 °C) to determine air temperature.

2.2.3. Calibrated hygrometer with sufficiently small resolution, stability, and uncertainty (see SOP 2, e.g., accurate to ± 10 percent) to determine relative humidity.

2.3. Estimating the Magnitude of the Air Buoyancy Correction

2.3.1. Estimate the magnitude of the air buoyancy correct, *MABC*, using the following formula:

$$MABC = (\rho_a - \rho_n)(V_x - V_s)$$

2.3.2. The equation may also be represented as follows:

$$MABC = m_o(\rho_a - \rho_n)\left(\frac{1}{\rho_x} - \frac{1}{\rho_s}\right)$$

Table 1. Variables for *MABC* equation.

Variable	Description of Variable
ρ_a	air density at the time of the measurement in mg/cm^3
ρ_n	density of "normal" air; i.e., 1.2 mg/cm^3
m_o	nominal mass (in grams)
V_x	volume of the unknown weight, *X* in cm^3
V_s	volume of the reference standard, *S* in cm^3
ρ_s	density of reference standard, *S* in g/cm^3
ρ_x	density of unknown weight, *X* in g/cm^3

The relative magnitude of the correction can be compared to the expanded measurement uncertainty to determine the importance of the air buoyancy correction for a particular measurement. In some mass calibration procedures, when the calculated value is sufficiently small compared to applicable tolerances, the value

[1] The barometer, thermometer, and hygrometer are used to determine the air density at the time of the measurement. The air density is used to make an air buoyancy correction. The accuracies specified are recommended for high precision calibration. Less accurate equipment can be used with only a small degradation in the overall accuracy of the measurement.

may be treated as an uncorrected systematic error and incorporated into the uncertainty calculations.

2.4. Procedure

2.4.1. Record the temperature, pressure, and relative humidity at the start and at the end of the measurement process as near the location of the measurement as necessary and practical. If these parameters change significantly during the measurement process, it may be necessary to wait for more stable operating conditions or to use average values to compute the air density. Use of the average environmental values may influence the uncertainty of the measurement result and must be evaluated for significance.

2.4.2. Determine the air density using the equation given in Section 8 of the Appendix to this SOP.

3. Calculations

3.1. Calculate the mass, M_x, of the unknown weight, X, using the following equation, where d represents the "difference" obtained with buoyancy corrections applied to the sensitivity weight.

$$M_x = \frac{M_s\left(1 - \frac{\rho_a}{\rho_s}\right) + d}{\left(1 - \frac{\rho_a}{\rho_x}\right)}$$

If tare weights were carried with X and/or S, use the following equation:

$$M_x = \frac{M_s\left(1 - \frac{\rho_a}{\rho_s}\right) + d + M_{t_s}\left(1 - \frac{\rho_a}{\rho_{t_s}}\right) - M_{t_x}\left(1 - \frac{\rho_a}{\rho_{t_x}}\right)}{\left(1 - \frac{\rho_a}{\rho_x}\right)}$$

Table 2. Variables not previously defined.

Variable	Description of Variable
d	measured difference between X and the reference standard, S, using one of the weighing designs given in other SOPs
M_S	[true] mass of the reference standard
M_{t_s}	[true] mass of the tare weights carried with S
M_{t_x}	[true] mass of the tare weights carried with X
ρ_S	density of the reference standard, S
ρ_X	density of the unknown standard, X
ρ_{t_s}	density of the tare weights carried with S
ρ_{t_x}	density of the tare weights carried with X

3.2. If reporting the conventional mass[2], CM_x, compute it using the following.

$$CM_x = \frac{M_x\left(1 - \frac{0.0012}{\rho_x}\right)}{\left(1 - \frac{0.0012}{8.0}\right)}$$

3.3. If reporting the apparent mass, AM_x, versus brass, compute it using the following.

$$AM_{x\,vs\,brass} = \frac{M_x\left(1 - \frac{0.0012}{\rho_x}\right)}{\left(1 - \frac{0.0012}{8.3909}\right)}$$

3.4. The conventional and apparent mass values are related by the following:

$$CM_x = \frac{AM_{x\,vs\,brass}\left(1 - \frac{0.0012}{8.3909}\right)}{\left(1 - \frac{0.0012}{8.0}\right)}$$

[2] Conventional Mass: "The conventional value of the result of weighing a body in air is equal to the mass of a standard, of conventionally chosen density, at a conventionally chosen temperature, which balances this body at this reference temperature in air of conventionally chosen density." The conventions are: reference density 8.0 g/cm^3; reference temperature 20 °C; *normal* air density 0.0012 g/cm3. Conventional mass was formerly called "Apparent Mass versus 8.0 g/cm^3" in the United States and is not recommended. *See OIML D28 (2004).*

4. Assignment of Uncertainty

The uncertainty in determining the air buoyancy correction is usually negligible relative to the precision of the measurement process itself. Consequently, the uncertainty for the measurement is based upon the uncertainty for the measurement process used and the uncertainty associated with the environmental measurement standards. The uncertainty in the CIPM[3] 2007 air density equation is 0.000 026 4 mg/cm^3 (or 0.002 2 % of normal air density.)

Table 3. Tolerances for measurements related to air density estimation.

	Uncertainty of air density values in % of air density		
Variable	± 0.1 % of air density	± 1.0 % of air density	Recommended (Section 2.2)
Air pressure (Pa)	± 101	± 1010	± 66.5
Air pressure (mm Hg)	± 0.76	± 7.6	± 0.5
Air temperature (°C)	± 0.29	± 2.9	± 0.1
Relative Humidity (%)	± 11.3	.....	± 10

[3] CIPM References: A Picard, R.S. Davis, M. Gläser, K. Fujii, Metrologia 45:149-155 (2008).

Appendix A

Based on "The Basic Theory of Air Buoyancy Corrections"

by Richard S. Davis[4]

1. Introduction

In performing measurements of mass, the balance or scale used acts as a force transducer. The force produced by an object to be weighed in air has two components: one proportional to the mass of the object, and the other proportional to its volume. The latter component, or buoyant force, may under some circumstances be large enough to require correction. The following shows under what circumstances buoyancy corrections are required as well as how they are made.

2. Scope

The method for applying buoyancy corrections presented below applies to mass measurements made in air. The density of air is computed from auxiliary measurements of temperature, pressure and relative humidity after which the buoyancy corrections are calculated directly from the Principle of Archimedes. The following weighing situations are considered.

2.1. Two-Pan Balance

2.2. Single-Pan Balance

2.2.1. With Built-In Weights

2.2.2. With Electronic Control

3. Summary of Method

In general, buoyancy corrections are applied to mass measurements by calculating the difference in volume between the unknown weight and the standard, multiplying this volume difference by the density of air at the balance or scale, and adding the product to the mass of the standard. The density of air is computed from an equation of state using measured values for the temperature, pressure and relative humidity of the air.

4. Significance and Use

Buoyancy corrections generally must be applied when determining the mass of an unknown object to high accuracy. The corrections may become important even at modest

[4]Richard S. Davis, formerly of the National Institute of Standards and Technology, Mass Group, International Bureau of Weights and Measures (BIPM), retired.

accuracies if the unknown object whose mass is to be determined has a density that differs widely from that of the standards (weighing of water, for example). Many mass standards are calibrated in terms of a so-called "apparent mass" [conventional mass] scale (See Chapter 7.3, Handbook 145[1]). Use of this scale does not indiscriminately eliminate the need for buoyancy corrections as is sometimes assumed.

5. Terminology

5.1. Weighing by Substitution

Substitution weighing is the procedure by which one determines the assembly of standard weights that will produce nearly the same reading on a one-pan balance as does the unknown object. The balance thus serves as a comparator. A two-pan balance may be used in this mode if one of the pans contains a counterpoise and the standards and unknown are substituted on the second pan. (See SOP No. 3.)

5.2. Mass

The term "mass" is always used in the strict Newtonian sense as a property intrinsic to matter. Mass is the proportionality constant between a force on a material object and its resulting acceleration. This property is sometimes referred to as "true mass", "vacuum mass", or "mass in vacuum" to distinguish it from conventional [apparent] mass.

5.3. Conventional [Apparent] Mass[5]

The mass of material of a specified density that would exactly balance the unknown object if the weighing were carried out at a temperature of 20 °C in air of density 0.0012 g/cm^3. The mass, M_N, of an object, N, is related to its apparent mass $M_{N,A}$ by the equation:

$$M_{N,A} = \frac{M_N \left(1 - \dfrac{0.0012}{\rho_N}\right)}{\left(1 - \dfrac{0.0012}{\rho_B}\right)}$$

[5]Pontius, P. E., Mass and Mass Values, NBS Monograph 122, 1974, pp 12-23, 26-33 described the concept of apparent mass. The term conventional mass is described by OIML D28, the Conventional Value of Mass in Air.

Table A-1. Variables for conventional (apparent) mass equation.

Variable	Description
ρ_N	density of the object N at 20 °C in g/cm^3
ρ_B	density of the conventional (apparent) mass scale at 20 °C in g/cm^3

There are at present two apparent mass scales in wide use. The older is based on ρ_B = 8.4000 g/cm^3 at 0 °C with a coefficient of volumetric expansion of 0.000054 /°C and the more recent (Conventional Mass) specifies ρ_B = 8.0000 g/cm^3 at 20 °C. The quantity $M_{N,A}$ is a function of the particular conventional or apparent mass scale, which has been used in its calculation. OIML D28 only recognizes Conventional Mass.

5.4. Sensitivity

The response of a balance under load to an additional small weight:

$$sensitivity = \frac{M_{sw} - \rho_a V_{sw}}{\Delta R} = \frac{M_{sw}\left(1 - \frac{\rho_a}{\rho_{sw}}\right)}{\Delta R}$$

Table A-2. Variables for sensitivity equation.

Variable	Description
sensitivity	balance sensitivity (mass per division)
M_{sw}	mass of the small, additional weight
ρ_a	density of the air
ρ_{sw}	density of the small, additional weight
V_{sw}	volume of the small, additional weight
ΔR	change in balance reading due to the addition of the small weight, balance deflection

6. Apparatus

In order to ascertain the density of air at the balance, the following measuring instruments are necessary: thermometer, barometer, and hygrometer. Ideally, these instruments should be placed in or next to the balance case (as near the measurement location as is practical). It may only be practical for the thermometer or temperature sensor to actually be placed inside the balance chamber. A calculator or computer will be extremely useful for this procedure.

7. Procedure

Weigh the unknown object as directed by the balance manufacturer or in accordance with accepted procedure. Record the temperature, pressure and relative humidity of the air in the balance at the time of weighing. Do not correct the barometric pressure to sea level.

8. Calculation

8.1 Air density, Option A (Option B is preferred)

The density of air, in g/cm^3, can be *approximated for lesser accuracy* from the following formula:[6]

$$\rho_a = \left\{ \frac{0.464\,60\left[P - \left(0.003\,796\,0\ U\ e_s \right) \right]}{\left(273.15 + t \right)} \right\} \text{x}\,10^{-3} \qquad (1)$$

Table A-3. Variables for air density equation.

Variable	Description
ρ_a	density of air, g/cm^3
P	barometric pressure, mm Hg
U	% relative humidity, entered as a whole number
t	temperature, °C
e_s	$1.314\,6 \text{ x } 10^9 \text{ x } e^{[-5\,315.56/(t + 273.15)]}$

Note: e_s can be written as follows in a spreadsheet and in some calculators:
1.3146E9*@EXP(-5315.56/(t+273.15))

Small errors (of order 0.01 %) in this equation occur for locations well above sea level or under conditions in which the concentration of carbon dioxide differs greatly from the global average. See the references for a more general formulation of the equation.

[6]Jones, F.E., "The Air Density Equation and the Transfer of the Mass Unit," Journal of Research, National Institute of Standards and Technology, Vol. 83, 1978, p. 419.

8.2. Air density, Option B – Preferred

The density of air should be calculated with the following formula.[7]

$$\rho_a = \frac{pM_a}{ZRT}\left(1 - x_v\left(1 - \frac{M_v}{M_a}\right)\right)$$

$$x_v = (h/100)\, f\, \frac{\rho_{sv}}{p}$$

$$Z = 1 - \frac{p}{T}\left[a_0 + a_1 t + a_2 t^2 + (b_0 + b_1 t)x_v + (c_0 + c_1 t)x_v^2\right] + \frac{p^2}{T^2}\left(d + e x_v^2\right)$$

Table A-4. Variables for CIPM air density equation.

Variable	Description
M_a	molar mass of the air within laboratory 28.965 46 x 10^{-3} kg/mol
M_v	18.01528(17) x10^{-3} kg m^{-3}
p	ambient barometric pressure in Pascal
T	ambient temperature in Kelvin
R	universal gas constant: 8.314 472 J mol^{-1} K^{-1}
h	relative humidity in %
f	1.000 62 + (3.14 x 10^{-8}) p + (5.6 x 10^{-7})t^2
t	ambient temperature in degrees Celsius
p_{sv}	1 Pascal x exp (AT^2 + BT + C + D/T)
A	1.237 884 7 x 10^{-5} K^{-2}
B	-1.912 131 6 x 10^{-2} K^{-1}
C	33.937 110 47
D	-6.343 164 5 x 10^3 K
a_0	1.581 23 x 10^{-6} K Pa^{-1}
a_1	-2.933 1 x 10^{-8} Pa^{-1}
a_2	1.104 3 x 10^{-10} K^{-1} Pa^{-1}
b_0	5.707 x 10^{-6} K Pa^{-1}
b_1	-2.051 x 10^{-8} Pa^{-1}
c_0	1.989 8 x 10^{-4} K Pa^{-1}
c_1	-2.376 x 10^{-6} Pa^{-1}
d	1.83 x 10^{-11} K^2 Pa^{-2}
e	-0.765 x 10^{-8} K^2 Pa^{-2}

[7] CIPM References: Giacomo, P. Metrologia 18: 33-40 (1982), Davis, R.S., Metrologia 29: 67-70 (1992), A Picard, R.S. Davis, M. Gläser, K. Fujii, Metrologia 45:149-155 (2008).

Calculate the density of air at the balance during the weighing. Then determine the mass of the unknown, M_x, as follows:

8.3 If a two-pan balance is used:

$$M_x = M_s + \rho_a \left(V_x - V_s \right) + sensitivity\, \Delta M_{opt} \qquad (3a)$$

or

$$M_x = \frac{M_s \left(1 - \frac{\rho_a}{\rho_s} \right) + sensitivity\, \Delta M_{opt}}{\left(1 - \frac{\rho_a}{\rho_x} \right)} \qquad (3b)$$

or

$$M_x = \frac{M_s - \rho_a V_s + sensitivity\ \Delta M_{opt}}{\left(1 - \frac{\rho_a}{\rho_x} \right)} \qquad (3c)$$

Table A-5. Variables not previously defined.

Variable	Description
M_x	mass of the unknown object
M_s	mass of the standard weights
ρ_s	density of the standard weights, $\frac{M_S}{V_S}$
ΔM_{opt}	off-balance indication read on the optical scale
V_x	volume of the unknown object
V_s	volume of the standard weights
ρ_x	density of the unknown object, $\frac{M_X}{V_X}$

Volumes and densities are, in general, a function of temperature. The thermal coefficients of volumetric expansion of the unknown object and the standard may be significant in very accurate work.[8] The coefficient of volumetric expansion is

[8]In general, $V_t = V_{20}\,[1 + ß(t - 20\ °C)]$ where t is the temperature of the weight, V_{20} is the volume at 20 °C, and ß is the cubical coefficient of expansion.

usually estimated as three times the linear coefficient of expansion of the weight material.

The error in M_x incurred by ignoring the buoyancy correction is $\rho_a (V_x - V_s)$. To estimate quickly whether such an error is of consequence in a particular measurement, (assume $\rho_a = 1.2 \times 10^{-3} g/cm^3$).

If the mass and volumes of the standards have been adjusted to a conventional mass scale, then

$$M_x \approx \frac{CM_s \left(1 - \frac{\rho_a}{\rho_B}\right) + sensitivity\, \Delta M_{opt}}{\left(1 - \frac{\rho_a}{\rho_x}\right)} \qquad (4)$$

Table A-6. Variables not previously defined.

Variable	Description
CM_s	conventional mass of the standard
The symbol ≈ signifies an approximation	

8.4 If a single-pan balance with built-in weights is used, it is probable that the built-in weights have been adjusted on an apparent mass or conventional mass scale[9]. Determine which apparent mass scale has been used and calculate the mass of the unknown from the equation

$$M_x \approx \frac{M_D \left(1 - \frac{\rho_a}{\rho_B}\right) + sensitivity\, M_{opt}}{\left(1 - \frac{\rho_a}{\rho_x}\right)} \qquad (5)$$

Table A-7. Variables not previously defined.

Variable	Description
M_D	mass indicated by dial or digital readings
M_{opt}	mass indicated on the optical scale when present

[9]Schoonover, R. M. and Jones, F. E., "Air Buoyancy in High-Accuracy Weighing on Analytical Balances," Anal. Chem., 53, 1981, p. 900.

If the balance has been used only as a comparator, that is, to compare the mass of the unknown object with that of some external standard, then:

$$M_x = M_s + \rho_a \left(V_x - V_s \right) + sensitivity\, \Delta M'_{opt}$$

Table A-8. Variable not previously defined.

Variable	Description
$\Delta M'_{opt}$	difference in optical scale reading between observations of the standard and the unknown

For some balances, operation requires that the user restore the balance to null by means of a manually controlled dial. The portion of the mass reading controlled by this dial should be treated, for purposes of buoyancy corrections, as an optical scale.

8.5 If a single-pan balance with full-range electronic control is used, the following should be noted. As part of its calibration, the electronic gain has been adjusted by means of a weight of known mass. For example, if the range of electronic control is 100 g, the electronics have been adjusted so that a 100 g standard weight produces an indication of precisely 100 g. This procedure effectively builds an apparent mass calibration into the balance. The reference density of the apparent mass scale is the density of the standard mass used for the calibration and the reference air density is the air density at the time of calibration.

The mass of an unknown object weighed on the balance is then

$$M_x = \frac{M_R \left(1 - \dfrac{\rho_a}{\rho_c} \right)}{\left(1 - \dfrac{\rho_a}{\rho_x} \right)} \qquad (6)$$

Table A-9. Variables not previously defined.

Variable	Description
M_R	readout displayed on the balance
ρ_a	density of air at the time of balance calibration
ρ_c	density of the standard used to calibrate the balance (or ρ_B if the apparent [conventional] mass of the standard was used instead of the true mass

If the balance includes both an electronic control system and built-in weights, the buoyancy considerations for the built-in weights are as described in section 8.2 and the considerations for the electronically determined mass are those given directly above.

8.6 Top-loading balances may be considered a form of single-pan balance and the appropriate procedure for buoyancy correction followed.

9. Precision

The contribution of the random error of the evaluation of air density to the precision of mass measurement may be estimated as follows:

For mechanical balances, or electronic balances used in weighing by substitution, the contribution is:

$$\delta\rho_a \left(V_x - V_s \right)$$

Table A-10. Variables for above equation.

Variable	Description
$\delta\rho_a$	random error of evaluation of ρ_a
V_s	volume of standards, if weighing by substitution $V_s = M_D / D_B$, if using the built-in weights on a single pan balance.
V_x	volume of object weighed

The quantity, $\delta\rho_a$ will have contributions from the measurements of temperature, pressure and relative humidity which are required for the calculation of ρ_a. Equation (1) may be used to estimate the effects of imprecision in measurements of P, t, and U. It is unrealistic to expect $\delta\rho_a / \rho_a$ ever to be less than 0.05 % even using the best techniques available.

10. Accuracy

Inattention to problems of buoyancy most often results in systematic errors. For a substitution weighing, for example, the buoyancy correction is of the order of $\rho_a(V_x - V_s)$. While this quantity may be significant to a measurement, day-to-day variation in ρ_a (usually no more than 3 %) may not be noticeable and hence need not be measured. For the most accurate work, not only must ρ_a be accurately determined, but the volumes of the unknown and standard may have to be measured to better than 0.05 % - the minimum systematic uncertainty attainable in the calculation of ρ_a.

If the standards have been calibrated in terms of conventional mass, complete neglect of buoyancy corrections will produce an error in the measured result M_x of order:

$$CM_s \left[\left(1.2 \, x 10^{-3} - \rho_a \right) \left(\frac{1}{\rho_s} - \frac{1}{\rho_B} \right) + \left(\frac{\rho_B - \rho_x}{\rho_B} \right) \left(\frac{\rho_a}{\rho_x} \right) \right] \qquad (7)$$

This error is often unacceptably large.

Use of equation (4), on the other hand, introduces only an error of approximately

$$CM_s \left(1.2\, x\, 10^{-3} - \rho_a\right)\left(\frac{1}{\rho_s} - \frac{1}{\rho_B}\right) \qquad (8)$$

It is a requirement for manufacture that the actual density of standard weights be near enough to the assumed density of the apparent mass scale to which they are adjusted that the magnitude of (8) will always be small under normal conditions in laboratories near sea level.

The fact that there are two apparent mass scales widely used - one based on density 8.0 g/cm^3 and an older one based on 8.4 g/cm^3 - means that some caution is required on the part of the user. Conventional mass is generally preferred and reported for all calibrations where mass standards will be used to calibrate weighing instruments. For the most accurate work, the apparent mass scale should be abandoned in favor of substitution weighing with standards of known mass, density, and volume.

The user must decide the accuracy required of the particular mass measurement and choose a buoyancy correction technique commensurate with that accuracy.

The same considerations, which apply to the accuracy of buoyancy corrections in weighing by substitution, are easily extended to the other types of weighing indicated above.

There are many factors, which affect the accuracy of a mass measurement. The above has dealt only with those arising from problems of buoyancy.

Appendix B

Examples

Example 1:

The weight set of Table B-1 is used with an equal-arm balance to find the mass of a piece of single-crystal silicon. The following weights were used to balance the silicon: 10 g, 3 g. The balance pointer showed the silicon side to be light by 3.5 divisions. The 10 mg weight of Table 1 was used to find the sensitivity of the balance. When the weight was added, the pointer moved 10.3 divisions. At the time of the weighing, the following measurements were taken:

P = 748.1 mm Hg

t = 22.3 ℃

U = 37 % relative humidity

What is the mass of the silicon?

Answer:

From Eq. 1, or Table B-2, calculate

$$e_s = 1.3146 \, x 10^9 \; x \; e^{\left(\frac{-5315.56}{(22.3 + 273.15)}\right)} = 20.194 \text{ mm Hg}$$

$$\rho_a = \left\{ \frac{0.46460\left[748.1 - (0.0037960)(37)(20.2)\right]}{(273.15 + 22.3)} \right\} 10^{-3}$$

ρ_a = 1.171 94 x 10^{-3} g/cm^3

The density of silicon at 20 °C is 2.329 1 g/cm^3 and its coefficient of linear expansion is 0.000 002 6 /°C.

Make use of Eq. 3c and Table B-1.

$$M_s = 10.0001264 + 3.0000459 = 13.000172 \text{ g}$$

$$V_s = (1.267 + 0.380) = 1.647 \text{ cm}^3$$

Calculate the sensitivity:

$$\text{Sensitivity} = \frac{0.010\,003\,g - (0.003\,70)(1.172\,x10^{-3})}{10.3}\ \text{g/division}$$

$$= \frac{(0.009\,999)}{10.3} = 0.000\,971\,\text{g/division} = 0.971\ \text{mg/division}$$

$$M_x = \frac{\left[13.000\,172 - (1.647)(1.172\,x10^{-3})\right] + \left[(0.971\,x10^{-3})(-3.5)\right]}{\left(1 - \frac{1.172\,x10^{-3}}{2.329\,1}\right)}$$

M_x = 13.001 385 g

Note that the thermal expansion is insignificant in this example.

Example 2:

Let us again consider the weighing performed in Example 1. This time, all we know about our weight set is that it has been adjusted to the 8.4 apparent mass scale at the best available commercial tolerance.

Using Eq. (4),

$$M_x = \left\{ \frac{\left[13.00\left(1 - \frac{1.172\,x10^{-3}}{8.390\,9}\right)\right] + \left[0.010\left(1 - \frac{1.172\,x10^{-3}}{8.390\,9}\right)\right]\left(\frac{-3.5}{10.3}\right)}{\left(1 - \frac{1.172\,x10^{-3}}{2.329\,1}\right)} \right\}$$

M_x = 13.001 329 g

For routine weighing, it is sometimes satisfactory to assume that the temperature is 20 °C and the density of air is 1.2 x 10^{-3} g/cm^3. Had this been done, the computed value for the silicon would be

$$M_x = \left\{ \frac{\left[13.00\left(1 - \frac{1.2\,x10^{-3}}{8.390\,9}\right)\right] + \left[0.010\left(1 - \frac{1.2\,x10^{-3}}{8.390\,9}\right)\right]\left(\frac{-3.5}{10.3}\right)}{\left(1 - \frac{1.2\,x10^{-3}}{2.329\,1}\right)} \right\}$$

M_x = 13.001 442 g

which is within 100 µg of the answer found in Example 1.

Example 3:

Another piece of silicon is measured on a single-pan microbalance. The balance weights were adjusted by the manufacturer to the conventional mass scale. The sensitivity of the balance has been determined to be exactly 1.000. This particular laboratory is well above sea level. At the time of the weighing, the following measurements were recorded:

P = 612.3 mm Hg

t = 23.4 °C

U = 23 % relative humidity

The balance reading was 15.00 g on the built-in weights and 0.000 358 g on the optical screen. What is the mass of the silicon?

Answer:

First, calculate e_s and ρ_a:

e_s = 21.59 mm Hg

$\rho_a = 0.956 \times 10^{-3}$ g/cm^3

Then, use Eq. 5:

$$M_x = \frac{(15.00 + 0.000\,358)\left(1 - \dfrac{0.956\,x\,10^{-3}}{8.0}\right)}{\left(1 - \dfrac{0.956\,x\,10^{-3}}{2.3291}\right)}$$

M_x = 15.004 724 g

Example 4:

The built-in weights in Example 3 are actually stainless steel of density 7.78 g/cm^3 at 20 °C. What is the approximate error caused by using the apparent mass scale?

Answer:

Using (7), the error is approximately

$$15\ g\left(0.2\ x\,10^{-3}\ g/cm^3\right)\left(\frac{1}{7.78\ g/cm^3}-\frac{1}{8.0\ g/cm^3}\right)=11\ \mu g$$

This discrepancy, though larger than the precision of the best analytical balances, is actually well within the tolerance of ASTM Class 1 weights.

Table B-1. Example of calibration report data.

Mass (g)	Uncertainty (g)	Vol at 20 °C (cm^3)	Cubical Coefficient of Expansion (/°C)
100.000 941	0.000 025	12.674 39	0.000 045
50.000 463	0.000 016	6.337 19	0.000 045
30.000 293	0.000 014	3.802 32	0.000 045
20.000 158	0.000 011	2.534 87	0.000 045
10.000 130	0.000 013	1.267 44	0.000 045
5.000 042	0.000 0069	0.633 72	0.000 045
3.000 046	0.000 0046	0.380 23	0.000 045
2.000 006 3	0.000 003 3	0.253 49	0.000 045
1.000 014 4	0.000 003 0	0.126 74	0.000 045
0.499 953 8	0.000 001 6	0.030 12	0.000 020
0.299 961 4	0.000 001 2	0.018 07	0.000 020
0.199 949 84	0.000 000 87	0.012 05	0.000 020
0.099 963 78	0.000 000 91	0.006 02	0.000 020
0.049 986 59	0.000 000 72	0.003 01	0.000 020
0.029 991 00	0.000 000 77	0.001 81	0.000 020
0.020 005 70	0.000 000 66	0.007 41	0.000 069
0.010 002 77	0.000 000 86	0.003 70	0.000 069
0.004 997 06	0.000 000 70	0.001 85	0.000 069
0.003 002 99	0.000 000 76	0.001 11	0.000 069
0.002 001 97	0.000 000 66	0.000 74	0.000 069
0.001 000 83	0.000 000 86	0.000 37	0.000 069

Table B-2. e_s approximation in terms of temperature.

Temperature (°C)	e_s (mm Hg)
18	15.48
18.5	15.97
19	16.48
19.5	17
20	17.54
20.5	18.09
21	18.65
21.5	19.23
22	19.83
22.5	20.44
23	21.07
23.5	21.72
24	22.38
24.5	23.07
25	23.77
25.5	24.49
26	25.23
26.5	25.99
27	26.77
27.5	27.57
28	28.39
28.5	29.23
29	30.09
29.5	30.98

[1] Handbook 145 is out of print as of 2012. Section 7 was updated and published as a part of NISTIR 6969 in 2003, 2012, and 2014.

SOP No. 4

Recommended Standard Operating Procedure for Weighing by Double Substitution

1. Introduction

1.1. Purpose

The double substitution procedure is one in which a standard and an unknown weight of equal nominal value are compared twice to determine the average difference between the two weights. Errors in any built-in weights or in the weighing instrument indications are minimized by using the weighing instrument as a comparator and by calibrating the weighing instrument indications over the range of use for the measurement with a sensitivity weight that has a known mass. Accordingly, the procedure is especially useful for calibrations of weights in OIML[1] Classes F_1 to M_3, ASTM[2] Classes 2 through 7, and forms the basis for advanced weighing designs used in SOP 5 and SOP 28 (See NISTIR 5672, 2014). The procedure incorporates two methods of measurement assurance: one is a mathematical check that evaluates the within-process repeatability and the other involves using a check standard to monitor the reference value(s) of the standard.

1.2. Prerequisites

1.2.1. Valid calibration certificates with appropriate values and uncertainties must be available for all of the standards used in the calibration. All standards must have demonstrated metrological traceability to the international system of units (SI), which may be to the SI through a National Metrology Institute such as NIST.

1.2.2. Standards must be evaluated to ensure that standard uncertainties for the intended level of calibration are sufficiently small. Reference standards should only be used to calibrate the next lower level of working standards in the laboratory and should not be used to routinely calibrate customer standards.

1.2.3. The weighing instrument must be in good operating condition with sufficiently small process standard deviation as verified by a valid control chart. When a new weighing instrument is put into service, preliminary

[1] OIML is the International Organization for Legal Metrology. Weight classes are published in OIML R 111, which is freely available at http://www.oiml.org.
[2] ASTM International (formerly the American Society for Testing and Materials) publishes the E617 standard for mass specifications and tolerances.

experiments must be conducted to ascertain initial performance quality and adequacy.

1.2.4. The operator must be trained and experienced in precision weighing techniques with specific training in SOP 2, SOP 4, SOP 29, GMP 4, and GMP 10.

1.2.5. Laboratory facilities must comply with the following minimum conditions to meet the expected uncertainty possible with this procedure and to comply with the balance manufacturer's operating conditions specified for the balance. Equilibration of balances and weights requires environmental stability of the laboratory within the stated limits for a minimum of 24 hours before a calibration.

Table 1. Environmental conditions.

Echelon[3]	Temperature Requirements During a Calibration	Relative Humidity (%)
II	Lower and upper limits: 18 °C to 23 °C Maximum changes: ± 2 °C / 12 h and ± 1.5 °C / h	40 to 60 ± 10 / 4 h
III	Lower and upper limits: 18 °C to 27 °C Maximum changes: ± 5 °C / 12 h and ± 3 °C / h	40 to 60 ± 20 / 4 h

1.2.5.1. Standards and test artifacts must be allowed to reach equilibration in or near the balance before commencing measurements. It is essential for the difference in temperature between the weights and the air inside the mass comparator to be as small as possible. Keeping the reference weight and the test weight inside, or next to, the mass comparator before and during the calibration can reduce this temperature difference.

2. Methodology

2.1. Scope, Precision, Accuracy

This method is applicable for all types of weighing instruments. The achievable precision and expanded uncertainty depends on the resolution, sensitivity, linearity, and other operating characteristics of the weighing instrument and the care exercised to make the required weighings. The accuracy achievable with this procedure depends on the accuracy of the calibration of the working standards and the accuracy obtained in the measurement process.

[3] Echelon II corresponds to weights of Classes OIML F_1 and F_2. Echelon III corresponds to weights of Classes OIML M_1, M_2, and M_3. This procedure does not provide adequate redundancy for OIML Classes E_1 or E_2 in which case SOP 5 or SOP 28 should be used.

2.2. Summary

The weighing instrument is adjusted if necessary, to obtain measurement indications for all weighings that are within the range of the optical scale or digital indications without changing built-in weights, if present (this includes changing dials or the weighing platform masses). The standard and the test weight are each weighed twice. A small, calibrated weight, called a sensitivity weight, is added to the standard and unknown test weight in the latter two weighings to provide a determination of the sensitivity of the balance under the load conditions at the time of the intercomparison. All weighings are made at regularly spaced time intervals to minimize effects due to drift in the measurement process.

The double substitution procedure is the same for all weighing instruments, but the adjustment of the weighing instrument to prepare for the intercomparison and the selection of the sensitivity weight differs slightly depending upon the type of instrument used. When steps specific to a particular instrument are required, they are given in subsections of the procedure identified by a, b, and c along with the type of instrument.

2.3. Apparatus/Equipment Required

2.3.1. Precision analytical balance or mass comparator with sufficient capacity and resolution for the calibrations planned.

2.3.2. Calibrated working standards, of nominally equal mass to the unknown mass standards being calibrated. Calibrated tare weights are used as needed to ensure that the standard(s) and test artifacts are of equal nominal mass (See SOP 34 for suitable limits).

2.3.3. Calibrated sensitivity weights and tare weights selected to comply with the requirements of SOP 34.

2.3.4. Uncalibrated weights to be used to adjust the balance to the desired reading range if needed.

2.3.5. Forceps to handle the weights, or gloves to be worn if the weights are moved by hand. Forceps and gloves must be selected to avoid damage or contamination of mass standards.

2.3.6. Stop watch or other timing device to observe the time of each measurement (calibration not required; this is used to ensure consistent timing of the measurement). If an electronic weighing instrument is used that has a means for indicating a stable reading, the operator may continue to time readings to ensure consistent timing that can minimize errors due to linear drift.

2.3.7. Calibrated barometer with sufficiently small resolution, stability, and uncertainty (See SOP 2, e.g., accurate to ± 66.5 Pa (0.5 mm Hg)) to determine barometric pressure. [4]

2.3.8. Calibrated thermometer with sufficiently small resolution, stability, and uncertainty (see SOP 2, e.g., accurate to ± 0.10 °C) to determine air temperature.[3]

2.3.9. Calibrated hygrometer with sufficiently small resolution, stability, and uncertainty (see SOP 2, e.g., accurate to ± 10 percent) to determine relative humidity.[3]

2.4. Symbols

Table 2. Symbols used in this procedure.

Symbol	Description
S	standard reference weight
X	weight to be calibrated
S_c	check standard
t	small calibrated tare weight, A subscript *s* or *x* is used to indicate the larger weight with which it is associated
sw	small calibrated weight used to evaluate the sensitivity of the balance
M	the mass (true mass) of a specific weight. Subscripts *s*, *x*, *t*, *sw* are used to identify the weight (equals Nominal plus Correction)
N	the nominal value of a specific weight. Subscripts *s*, *x*, are used to identify the weight.
C	the correction for a specific weight. Subscripts *s*, *x*, are used to identify the weight.
CM	the conventional mass of a specific weight. Subscripts *s*, *x*, *t*, *sw* are used to identify the weight.
ρ_a	density of air at time of calibration
ρ_n	density of normal air (1.2 kg/m^3)
ρ	density of masses; subscripts *s*, *x*, t_s, t_x, *sw* are used to identify the weight.

[4]The barometer, thermometer, and hygrometer are used to determine the air density at the time of the measurement. The air density is used to make an air buoyancy correction. The limits specified are recommended for high precision calibration.

2.5. Procedure

2.5.1. Preliminary Procedure

2.5.1.1. Weights are visually inspected for cleanliness and damage. Follow the laboratory policy, including discussions with customers, to determine if and when standards will be cleaned, or where standards with inadequate cleanliness are returned without calibration and when “as found” and “as left” values will be obtained through duplicate calibrations.

2.5.1.2. If cleaning weights, it is important to clean weights before any measurements are made, unless “as found’ data is to be measured, because the cleaning process will likely change the mass of the weight. Cleaning should not remove any significant amounts of weight material. Weights should be handled and stored in such a way that they stay clean. Before calibration, dust and any foreign particles shall be removed by blowing air across the surface with a clean bulb or by brushing with a clean soft bristled brush. Care must be taken not to change the surface properties of the weight (i.e., by scratching the weight). If a weight contains significant amounts of dirt that cannot be removed by the methods cited above, the weight or some part of it can be washed with clean alcohol, distilled water or other solvents. Weights with internal cavities should normally not be immersed in the solvent to avoid the possibility that the fluid will penetrate the opening. If there is a need to monitor the stability of a weight in use, the mass of the weight should, if possible, be determined before cleaning.

2.5.1.3. If weights are cleaned with solvents they must be stabilized for the times given in the following table (better class weights need to stabilize for 7 to 10 days):

Table 3. Cleaning stabilization.

Weight class	F_1	F_2 to M_3
After cleaning with alcohol	1 to 2 days	1 hour
After cleaning with distilled water	1 day	1 hour

2.5.1.4. Prior to performing any calibration tests, the weights need to be equilibrated to the ambient conditions of the laboratory. In particular, weights of classes F_1 (or better) should be close to the temperature in the weighing area. The minimum times (in hours)

required for temperature stabilization (depending on weight size, weight class and on the difference between the initial temperature of the weights and the room temperature in the laboratory) are shown in the table below (with appropriate documented evidence). As a practical guideline, a waiting time of 24 hours is recommended. If weights are extremely hot or cold, additional equilibration is required to address problems with varying surface moisture levels. Weights must be completely dry prior to calibration.

Table 4. Minimum equilibration times[5].

ΔT*	Nominal Mass	OIML Class F_1 (time in h)	OIML Class F_2 to M_3 (time in h)
± 20 °C	1 000, 2 000, 5 000 kg	79	5
	100, 200, 500 kg	33	4
	10, 20, 50 kg	12	3
	1, 2, 5 kg	6	2
	100, 200, 500 g	3	1
	10, 20, 50 g	1	1
	< 10 g	1	0.5
± 5 °C	1 000, 2 000, 5 000 kg	1	1
	100, 200, 500 kg	2	1
	10, 20, 50 kg	4	1
	1, 2, 5 kg	3	1
	100, 200, 500 g	2	0.5
	< 100 g	1	0.5
± 2 °C	< 100 g to 5 000 kg	1	0.5
± 0.5 °C	< 100 g to 5 000 kg	0.5	0.5

*ΔT = Initial difference between weight temperature and laboratory temperature.

[5] Consider equivalent ASTM Classes for equilibration times.

2.5.1.5. Conduct preliminary measurements to obtain an approximate value for the difference between the standard and the unknown, to identify where in the weighing instrument range the readings occur, to determine if tare weights are required, to determine the sensitivity weight to be used, and to determine the time interval required for the weighing instrument indication to stabilize. See NISTIR 6969, SOP 34 for specific instructions on evaluation of the need for and selection of tare weights and sensitivity weights.

Tare weights are rarely needed for mass standards that are within applicable tolerances. When unequal nominal weights are compared, tare weights are often required. When tare weights are required, carry tare weights, t_s and t_x, with the standard and the unknown, S and X, respectively. The tare weights must be calibrated standards with valid uncertainties that are included in the process of determining mass values and calibration uncertainties. The standard and its tare weight, $S + t_s$, should be "nearly the same mass" as the unknown with its tare weight, $X + t_x$. "Nearly the same mass" depends upon the balance used (See SOP 34, Table 1).

A sensitivity weight must be used on equal-arm balances, single-pan mechanical, and is usually used on electronic balances, to ensure that the measured differences between the standard(s) and unknown test items have valid accuracy and traceability (See SOP 34, Table 2) (e.g., the optical/digital scale is *calibrated* each time the procedure is performed through the use of a sensitivity weight).

If the sensitivity of the balance has been analyzed and is periodically evaluated and found to introduce negligible errors or uncertainties according to SOP 34 and measurement requirements, the equations in Section 3 must be modified to eliminate the sensitivity factor to avoid “division by zero” errors in the calculations.

If a sensitivity weight will be used, select one that meets the criteria in SOP 34.

2.5.1.6. Determine whether optional sequence A or B will be used. Optional sequence A uses the standard on the balance for the first and fourth observations and the unknown on the balance for the second and third observations; this is often called the “*SXXS*” sequence. Optional sequence B starts with the unknown

on the balance first and last with the standard on the balance for the second and third observations; this is often called the "*XSSX*" sequence. The primary advantage of sequence B is less handling of the mass standards. The advantage of sequence A is in the case where the unknown is a summation of weights that require careful arrangement on the balance pan only once. Option A is used in SOP 5 and SOP 28.

2.5.1.7. Adjust the single pan mechanical balance or the combination balance so the first two readings of the double substitution fall in the first quarter of the optical scale or digital indications. The zero adjustment and tare adjustment may be used. Small weights may be placed on the balance pan to reach the desired reading range. These weights remain on the pan throughout the double substitution and calibration is not required. Once the balance has been adjusted to the desired scale indication, neither the balance dials, the zero and tare adjustments, nor the small weights placed on the balance pan, are to be changed during the measurement.

2.5.1.8. If the balance is equipped with a pan arrestment mechanism, arrest the pan between each observation.

2.5.2. Measurement Procedure, Optional Sequence A (*SXXS*)

Table 5. Optional sequence A.

Measurement No.	Weights on Pan	Observation
1	$S + t_s$	O_1
2	$X + t_x$	O_2
3	$X + t_x + sw$	O_3
4	$S + t_s + sw$	O_4

All observations should be recorded on suitable data sheets such as those in the appendix or may be entered into computer software. Measure and record the laboratory ambient temperature, barometric pressure, and relative humidity before and after the mass measurements.

2.5.2.1. Observation 1. Place the standard weight(s), S, along with t_s (if applicable) on the balance pan. If equipped with a pan arrestment mechanism, release the balance pan. When the pan is released, time the interval or wait for the stability indicator and record observation O_1 once the balance indication has stabilized.

2.5.2.2. Observation 2. Remove weight(s) S and t_s (if present) and replace with test weight X and its tare weight, t_x (if applicable.) Release the pan, time the interval or wait for the stability indicator, and record observation O_2.

2.5.2.3. Observation 3. Add the sensitivity weight, *sw*, to the weights of observation 2. Pick up the largest weight on the pan to ensure consistent stabilization timing when needed. Release the pan, time the interval or wait for the stability indicator, and record observation O_3.

2.5.2.4. Observation 4. Remove weights X and t_x (if present) and replace with S and t_s (if applicable) on the balance pan. The sensitivity weight, *sw*, remains on the balance pan. Release the pan, time the interval or wait for the stability indicator, and record observation O_4.

2.5.2.5. Compare the two differences ($O_2 - O_1$) and ($O_3 - O_4$) to evaluate within process repeatability; they should not differ from one another by a laboratory determined limit (e.g., 2 standard deviations of the balance or 10 balance divisions are commonly used) for this process and load. If this difference is exceeded, reject the data and redo the measurements. Investigate possible causes of excess variability should within process repeatability exceed the limits.

2.5.3. Measurement Procedure, Optional Sequence B (*XSSX*)

Table 6. Optional Sequence B.

Measurement No.	Weights on Pan	Observation
1	$X + t_x$	O_1
2	$S + t_s$	O_2
3	$S + t_s + sw$	O_3
4	$X + t_x + sw$	O_4

Measurements for Option B are made as described in Option A except that X, S, t_x, and t_s are interchanged.

3. Calculations

3.1. If no air buoyancy correction is performed, calculate the conventional mass correction, C_x, for the test weight using the appropriate equation from 3.1.1 or 3.1.2, depending on the sequence used, and incorporate an uncorrected systematic uncertainty in the uncertainty calculations when using these equations due to the lack of performing air buoyancy corrections. Use the equations provided in SOP 2 to calculate the magnitude of the air buoyancy correction and treat it as a rectangular distribution.

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3.1.1. Optional Sequence A (*SXXS*)

$$C_x = C_s + CM_{t_s} - CM_{t_x} + \left[\frac{(O_2 - O_1) + (O_3 - O_4)}{2} \right] \left[\frac{CM_{sw}}{O_3 - O_2} \right] + N_s - N_x$$

3.1.2. Optional Sequence B (*XSSX*)

$$C_x = C_s + CM_{t_s} - CM_{t_x} + \left[\frac{(O_1 - O_2) + (O_4 - O_3)}{2} \right] \left[\frac{CM_{sw}}{O_3 - O_2} \right] + N_s - N_x$$

3.2. Mass Calculation with Air Buoyancy Correction

3.2.1. Calculate the air density, ρ_a, as described in the Appendix to SOP No. 2, Option B.

3.2.2. Calculate the mass M_{Sc} of the check standard using the mass of the standard weight(s), the tare weights, and the sensitivity weights, according to the optional sequence used.

3.2.2.1. Optional Sequence A (*SXXS*)

$$M_{Sc} = \frac{M_s\left(1 - \frac{\rho_a}{\rho_s}\right) + M_{t_s}\left(1 - \frac{\rho_a}{\rho_{t_s}}\right) - M_{t_x}\left(1 - \frac{\rho_a}{\rho_{t_x}}\right) + \left[\frac{(O_2 - O_1) + (O_3 - O_4)}{2}\right]\left[\frac{M_{sw}\left(1 - \frac{\rho_a}{\rho_{sw}}\right)}{O_3 - O_2}\right]}{\left(1 - \frac{\rho_a}{\rho_x}\right)}$$

3.2.2.2.Optional Sequence B (XSSX)

$$M_{Sc} = \frac{M_s\left(1 - \frac{\rho_a}{\rho_s}\right) + M_{t_s}\left(1 - \frac{\rho_a}{\rho_{t_s}}\right) - M_{t_x}\left(1 - \frac{\rho_a}{\rho_{t_x}}\right) + \left[\frac{(O_1 - O_2) + (O_4 - O_3)}{2}\right]\left[\frac{M_{sw}\left(1 - \frac{\rho_a}{\rho_{sw}}\right)}{O_3 - O_2}\right]}{\left(1 - \frac{\rho_a}{\rho_x}\right)}$$

3.2.3. Calculate the Conventional Mass[6] of S_c, CM_{Sc}.

$$CM_{Sc} = \frac{M_{S_c}\left(1 - \frac{\rho_n}{\rho_{Sc}}\right)}{\left(1 - \frac{\rho_n}{8.0}\right)}$$

3.2.4. Calculate the Mass Correction of S_c.

$$C_{Sc} = M_{Sc} - N_{Sc}$$

where N_{Sc} is the nominal value for *Sc* and ensure it is within statistical control or take suitable corrective action. The mass or conventional mass (depending on laboratory control charts) determined for the check standard should be plotted on the control chart and must lie within the control limits (see NISTIR 6969, SOP 9). If the value is not within limits, and the source of error cannot be found, measurement must be stopped until suitable corrective action is taken. Corrective action is demonstrated through evaluation of additional measurement results that are within limits.

[6] Conventional Mass: "The conventional value of the result of weighing a body in air is equal to the mass of a standard, of conventionally chosen density, at a conventionally chosen temperature, which balances this body at this reference temperature in air of conventionally chosen density." The conventions are: artifact reference density 8.0 g/cm^3; reference temperature 20 °C; *normal* air density 0.0012 g/cm^3. Conventional mass was formerly called "Apparent Mass versus 8.0 g/cm^3" in the United States. *See OIML D28 (2004).*

3.2.6 Calculate the mass of the test weight, M_x, and its mass correction, C_x, using the equations in 3.2.2.1 or 3.2.2.2 where S_c is replaced by X. Calculate the mass correction C_x, as follows:

$$C_x = M_x - N_x$$

where N_x is the nominal value for X.

3.2.7 Calculate the Conventional Mass[7] of X, CM_x. The conventional mass should be reported.

3.2.7.1. Conventional mass

$$CM_x = \frac{M_x \left(1 - \frac{\rho_n}{\rho_x} \right)}{\left(1 - \frac{\rho_n}{8.0} \right)}$$

3.2.8. If requested, the apparent mass versus the reference density of brass may be calculated. This value should only be provided when requested by the customer for use when calibrating mechanical balances that have been adjusted to this reference density. (This is rare.)

3.2.8.1. Apparent mass versus brass

$$AM_{x\ vs\ brass} = \frac{M_x \left(1 - \frac{\rho_n}{\rho_x} \right)}{\left(1 - \frac{\rho_n}{8.3909} \right)}$$

[7] Conventional Mass: "The conventional value of the result of weighing a body in air is equal to the mass of a standard, of conventionally chosen density, at a conventionally chosen temperature, which balances this body at this reference temperature in air of conventionally chosen density." The conventions are: reference density 8.0 g/cm^3; reference temperature 20 °C; *normal* air density 0.0012 g/cm3. Conventional mass was formerly called "Apparent Mass versus 8.0 g/cm^3" in the United States. *See OIML D28 (2004).*

4. Measurement Assurance

4.1. The process integrates a suitable check standard (See GLP 1, SOP 9, SOP 30, and Sec. 7.4).

4.2. The check standard value is calculated and immediately evaluated to verify that the mass is within established limits; a t-test may be incorporated to check the observed value against an accepted value.

4.3. The mean value of the check standard is compared to a reference value of the check standard with respect to their applicable expanded uncertainties to evaluate bias and drift over time. The mean value over time may be used to monitor drift in the standard or check standard. Excessive drift or bias must be investigated and followed with suitable corrective action.

4.4. Check standard observations obtained over time are used to calculate the standard deviation of the measurement process, s_p.

5. Assignment of Uncertainty

The limits of expanded uncertainty, U, include estimates of the standard uncertainty of the mass standards used, u_s, estimates of the standard deviation of the measurement process, s_p, and estimates of the effect of other components associated with this procedure, u_o. These estimates should be combined using the root-sum-squared method (RSS), and the expanded uncertainty, U, reported with a coverage factor to be determined based on the degrees of freedom, which if large enough will be 2, (k = 2), to give an approximate 95 percent level of confidence. See SOP 29 for the complete standard operating procedure for calculating the uncertainty.

5.1. The expanded uncertainty for the standard, U, is obtained from the calibration certificate. The combined standard uncertainty, u_c, is used and not the expanded uncertainty, U, therefore the reported uncertainty for the standard will usually need to be divided by the coverage factor k. When multiple standards are used, see SOP 29 for evaluation of dependent and independent conditions and combining methods for the standard uncertainty of the standard. Tare weights used in determining the mass of unknown weights are treated as additional standards in the measurement process.

Check standards are not used to assign values to unknown weights; however, the uncertainty of calibration of the check standard may be used to assess the control status.

5.2. The value for s_p is obtained from the control chart data for check standards using double substitution measurements. Where the standard deviation is less than the balance resolution, see section 5.2.1 and 5.2.2 to determine the smallest standard deviation to be included in the uncertainty calculations. Selection of 5.2.1 or

5.2.2 is left to the discretion of the technical manager based on the availability of data in the laboratory to ensure suitable confidence in measurement results. (See SOP No. 9.)

5.2.1. Where the standard deviation of the measurement process is less than the resolution of the balance being used, the larger of the s_p and the result from equation in SOP 29 may be used (e.g., this value may be estimated as 0.6 d).

$$u_d = \frac{d}{\sqrt{3}}$$

5.2.2. For SOP 4 and a digital balance with the scale interval, *d*, the smallest standard uncertainty due to resolution of the balance used in the measurement process may use:

$$u_d = \frac{d/2}{\sqrt{3}}$$

Because SOP 4 uses an average of two comparisons between S and X, the resolution, *d*, may be divided by 2. (It is not appropriate to use this minimum value in lieu of collecting suitable data for the measurement process.) E.g., this smallest value for the repeatability may be estimated as 0.3 *d*. The larger of the standard deviation of the measurement process over time or this value is to be used.

5.3. Uncertainty due to air buoyancy corrections and air density. Select one of the following options in priority preference for calculating and uncertainty associated with air buoyancy.

5.3.1. Option 1. Use the formulae provided in OIML R111 (2004), C.6.3-1, C.6.3-2, and C.6.3-3.

5.3.2. Option 2. Calculate the uncertainty by quantify estimated impacts associated with the uncertainties of the air temperature, barometric pressure, relative humidity, and the air density formula based on laboratory uncertainties and calculations given in NISTIR 6969, SOP 2 and the SOP being used. Note: this may be done using a simplified "baseline approach" or a Kragten analysis.[8]

5.3.3. Option 3. In the event that buoyancy corrections are not performed, include an uncorrected systematic standard uncertainty due to the

[8] A "baseline approach" calculates the estimated impact of each variable in the final measurement result by individually changing each variable of interest by the uncertainty quantity. See the EURACHEM/CITAC Quantitative Guide to Uncertainties in Analytical Methods (QUAM, 2012) for a discussion of Kragten spreadsheets.

buoyancy effect using the equations for the magnitude of the air buoyancy correction per SOP 2 and use a rectangular distribution. This approach is not recommended for precision calibrations; however, if the resulting value from this approach does not significantly affect the expanded uncertainty, it may be adequate.

5.4. Uncertainty associated with the density of the standards and the unknown test weights, u_{ρ}. Uncertainties associated with the density of the standards used in the calibration may be incorporated into the estimated calculations in section 5.3.

5.5. Uncertainty associated with bias, u_d. Any noted bias that has been determined through analysis of control charts and round robin data must be less than limits provided in SOP 29 and should be included if corrective action is not taken. See SOP 2 and 29 for additional details.

5.6. Example components to be considered for an uncertainty budget table are shown in the following table.

Table 6. Example uncertainty budget table.

Uncertainty Component Description	Symbol	Source	Typical Distribution
Uncertainty of the standard mass(es) (5.1)	u_s	Calibration certificate	Rectangular or Normal divided by coverage factor
Accepted standard deviation of the process (5.2)	s_p	Control chart, standard deviation chart	Normal
Uncertainty of the buoyancy correction (5.3)	u_b	OIML R111	Rectangular
Air temperature (for air density)	u_t	SOP 2 or R111	Rectangular
Air pressure (for air density)	u_p	SOP 2 or R111	Rectangular
Air relative humidity (for air density)	u_{RH}	SOP 2 or R111	Rectangular
Air density (formula)	$u_{\rho a}$	SOP 2 or R111	Rectangular
Mass densities (5.4)	$u_{\rho m}$	Measured and reported value OIML R111 Table B.7 Typically 0.03 g/cm^3 to 0.05 g/cm^3	Rectangular
Uncertainty associated with bias (5.5)	u_d	Control chart, proficiency tests	See SOP 29

5.7. Evaluate compliance to applicable tolerances as needed by the customer. The expanded uncertainty, U, must be $\leq 1/3$ of the applicable tolerances published in ASTM E617 and OIML R111 standards. Additionally, the mass value plus the expanded uncertainty must be less than the applicable tolerance to confidently state that mass standards are in or out of tolerance.

5.8. Draft a suitable uncertainty statement for the certificate.

For example: The uncertainty reported is the root sum square of the standard uncertainty of the standard, the standard deviation of the process, and an uncorrected systematic error for lack of buoyancy corrections, multiplied by a coverage factor of 2 ($k = 2$) for an approximate 95 percent confidence interval. Factors not considered in the evaluation: magnetism (weights are considered to meet magnetism specifications unless measurement aberrations are noted), balance eccentricity and linearity (these factors are considered as a part of the measurement process when obtaining the standard deviation of the process when using a check standard with adequate degrees of freedom).

6. Certificate

Report results as described in SOP No. 1, Preparation of Calibration Certificates.

Appendix
Double Substitution Data Sheet
(Optional Sequence A, *SXXS)*

Laboratory data and conditions:

			Before	After
Operator			Before	After
Date		Temperature		
Balance		Pressure		
Load		Relative Humidity		
Standard deviation of the process, from control chart, s_p		Degrees of Freedom		

Mass standard(s) data:

ID	Nominal	Mass Correction*	Expanded Unc: From cal. certificate	Unc: k factor	Density g/cm^3
S					
t_s					
X					
t_x					
S_c					
t_{Sc}					
sw					

*Mass Correction = *True Mass* if using buoyancy correction. Mass Correction = *Conventional Mass* if NOT using buoyancy correction. Density is used only with buoyancy corrections.

Observations:

Observation No.	Weights	Balance Observations, Units _____
Time:		
1 (O_1)	$S + t_s$	
2 (O_2)	$X + t_x$	
3 (O_3)	$X + t_x + sw$	
4 (O_4)	$S + t_s + sw$	
Time:		

Measurement Assurance (Duplication of the Process):

Observation No.	Weights	Balance Observations, Units _____
Time:		
1 (O_1)	$S + t_s$	
2 (O_2)	$S_c + t_{Sc}$	
3 (O_3)	$S_c + t_{Sc} + sw$	
4 (O_4)	$S + t_s + sw$	
Time:		
Check Standard in Control?	_____Yes_____No	

Appendix
Double Substitution Data Sheet
(Optional Sequence B, *XSSX)*

Laboratory data and conditions:

			Before	After
Operator			Before	After
Date		Temperature		
Balance		Pressure		
Load		Relative Humidity		
Standard deviation of the process, from control chart, s_p		Degrees of Freedom		

Mass standard(s) data:

ID	Nominal	Mass Correction*	Expanded Unc: From cal. certificate	Unc: k factor	Density g/cm^3
S					
t_s					
X					
t_x					
S_c					
t_{Sc}					
sw					

*Mass Correction = *True Mass* if using buoyancy correction. Mass Correction = *Conventional Mass* if NOT using buoyancy correction. Density is used only with buoyancy corrections.

Observations:

Observation No.	Weights	Balance Observations, Units
Time:		
1 (O_1)	$X + t_x$	
2 (O_2)	$S + t_s$	
3 (O_3)	$S + t_s + sw$	
4 (O_4)	$X + t_x + sw$	
Time:		

Measurement Assurance (Duplication of the Process):

Observation No.	Weights	Balance Observations, Units
Time:		
1 (O_1)	$S_c + t_{Sc}$	
2 (O_2)	$S + t_s$	
3 (O_3)	$S + t_s + sw$	
4 (O_4)	$S_c + t_{Sc} + sw$	
Time:		
Check Standard in Control?	_____Yes_____No	

SOP No. 7

Recommended Standard Operating Procedure for Weighing by Single Substitution Using a Single-Pan Mechanical Balance, a Full Electronic Balance, or a Balance with Digital Indications and Built-In Weights

1. Introduction

1.1. Purpose

In the single substitution procedure a standard and an unknown weight of equal nominal value are compared once to determine the difference in weights. Errors in any built-in weights or in the balance indications are minimized by using the balance only as a comparator and by calibrating the balance indications over the range of use for the measurement with a sensitivity weight. This procedure is suitable for calibration when moderate accuracy (OIML[1] Classes F_1 to M_3, ASTM[2] Classes 3 through 7) is required and as a single substitution, does not eliminate errors due to drift. The procedure incorporates measurement assurance through replicate tests using a check standard to monitor the reference value(s) of the standard and the repeatability of the measurement process over time.

1.2. Prerequisites

1.2.1. Valid calibration certificates with appropriate values and uncertainties must be available for all of the standards used in the calibration. All standards must have demonstrated metrological traceability to the international system of units (SI), which may be to the SI through a National Metrology Institute such as NIST.

1.2.2. Standards must be evaluated to ensure that standard uncertainties for the intended level of calibration are sufficiently small. Reference standards should not be used to routinely calibrate customer standards using this procedure.

1.2.3. Verify that the balance that is used is in good operating condition with sufficiently small process standard deviation as verified by a valid control chart or preliminary experiments to ascertain its performance quality when a new balance is put into service.

[1] OIML is the International Organization for Legal Metrology. Weight classes are published in OIML R 111, which is freely available at http://www.oiml.org.
[2] ASTM International (formerly the American Society for Testing and Materials) publishes the E617 standard for mass specifications and tolerances.

1.2.4. Verify that the operator is experienced in precision weighing techniques and has had specific training in SOP 2, SOP 7, SOP 29, GMP 4, and GMP 10.

1.2.5. Laboratory facilities must comply with the following minimum conditions to meet the expected uncertainty possible with this procedure and to comply with the balance manufacturer's operating conditions specified for the balance. The laboratory environment must be stable within the stated limits for a minimum of 24 hours before a calibration.

Table 1. Environmental conditions.

Echelon[3]	Temperature Requirements During a Calibration	Relative Humidity (%)
II	Lower and upper limits: 18 °C to 23 °C Maximum changes: ± 2 °C / 12 h and ± 1.5 °C / h	40 to 60 ± 10 / 4 h
III	Lower and upper limits: 18 °C to 27 °C Maximum changes: ± 5 °C / 12 h and ± 3 °C / h	40 to 60 ± 20 / 4 h

1.2.5.1. It is important that the difference in temperature between the weights and the air in the laboratory or inside the mass comparator be less than the values noted in the Procedure section. Standards and test artifacts must be allowed to reach equilibration in or near the balance before starting measurements.

2. Methodology

2.1. Scope, Precision, Accuracy

This method is applicable to all weighings utilizing a single-pan mechanical balance, a full electronic balance, or a balance that combines digital indications with the use of built-in weights (combination balance). The precision depends upon the sensitivity of the balance and the care exercised in making the required weighings. The accuracy achievable with this procedure depends on the accuracy of the calibration of the working standards and the precision of the intercomparison.

2.2. Summary

[3] Echelon II corresponds to weights of Classes OIML F_1 and F_2. Echelon III corresponds to weights of Classes OIML M_1, M_2, and M_3. This procedure does not provide adequate redundancy for OIML Classes E_1 or E_2 and SOP 5 or SOP 28 should be used.

The balance is adjusted, if necessary, to obtain balance indications for all measurements that will be within the range of the optical scale or digital indications of the balance without changing the dial settings for the built-in weights, if present. The standard and the test weight are each weighed. A small, calibrated weight, called a sensitivity weight, is added to the test weight and these are weighed

The single substitution procedure is the same for all of the balances mentioned above, but the adjustment of the balance to prepare for the intercomparison and the selection of the sensitivity weight differ slightly depending upon the balance used. When steps specific to a particular balance are required, they are given in subsections of the procedure identified by a, b, and c along with the balance type.

2.3. Apparatus/Equipment Required

2.3.1. Precision analytical balance or mass comparator with sufficient capacity and resolution for the calibrations planned.

2.3.2. Calibrated working standards, of nominally equal mass to the unknown mass standards being calibrated. Calibrated tare weights are used as needed to ensure that the standard(s) and test artifacts are of equal nominal mass (See SOP 34 for suitable limits).

2.3.3. Calibrated sensitivity weights and tare weights selected to comply with the requirements of SOP 34.

2.3.4. Uncalibrated weights to be used to adjust the balance to the desired reading range if needed.

2.3.5. Forceps to handle the weights, or gloves to be worn if the weights are moved by hand. Forceps and gloves must be selected to avoid damage or contamination of mass standards.

2.3.6. Stop watch or other timing device to observe the time of each measurement (calibration not required; this is used to ensure consistent timing of the measurement). If an electronic balance is used that has a means for indicating a stable reading, the operator may continue to time readings to ensure consistent timing that can minimize errors due to linear drift.

2.3.7. Calibrated barometer with sufficiently small resolution, stability, and uncertainty (See SOP 2, e.g., accurate to ± 66.5 Pa (0.5 mm Hg)) to determine barometric pressure.[4]

2.3.8. Calibrated thermometer with sufficiently small resolution, stability, and uncertainty (see SOP 2, e.g., accurate to ± 0.10 °C) to determine air temperature.[2]

2.3.9. Calibrated hygrometer with sufficiently small resolution, stability, and uncertainty (see SOP 2, e.g., accurate to ± 10 percent) to determine relative humidity.[2]

2.4. Symbols

Table 2. Symbols used in this procedure.

Symbol	Description
S	standard weight
X	weight calibrated
S_c	check standard
t	small calibrated tare weight, A subscript *s* or *x* is used to indicate the larger weight with which it is associated
sw	small calibrated weight used to evaluate the sensitivity of the balance
M	the mass (true mass) of a specific weight. Subscripts *s*, *x*, *t*, *sw* are used to identify the weight (equals Nominal plus Correction)
N	the nominal value of a specific weight. Subscripts *s*, *x*, are used to identify the weight.
C	the correction for a specific weight. Subscripts *s*, *x*, are used to identify the weight.
CM	the conventional mass of a specific weight. Subscripts *s*, *x*, *t*, *sw* are used to identify the weight.
ρ_a	density of air at time of calibration
ρ_n	density of normal air (1.2 kg/m^3)
ρ	density of masses; subscripts *s*, *x*, t_s, t_x, *sw* are used to identify the weight

[4]The barometer, thermometer, and hygrometer are used to determine the air density at the time of the measurement. The air density is used to make an air buoyancy correction. The limits specified are recommended for high precision calibration.

2.5. Procedure

2.5.1. Preliminary Procedure

2.5.1.1. Weights are visually inspected for cleanliness and damage.

2.5.1.2. If cleaning weights, it is important to clean weights before any measurements are made because the cleaning process may change the mass of the weight. Cleaning should not remove any significant amounts of weight material. Weights should be handled and stored in such a way that they stay clean. Before calibration, dust and any foreign particles shall be removed. Care must be taken not to change the surface properties of the weight (i.e. by scratching the weight). If a weight contains significant amounts of dirt that cannot be removed by the methods cited above, the weight or some part of it can be washed with clean alcohol, distilled water or other solvents. Weights with internal cavities should normally not be immersed in the solvent to avoid the possibility that the fluid will penetrate the opening. If there is a need to monitor the stability of a weight in use, the mass of the weight should, if possible, be determined before cleaning.

2.5.1.3. If weights are cleaned with solvents they must be stabilized for the times given in the following table (better class weights need to stabilize for 7 to 10 days):

Table 3. Cleaning stabilization.

Weight class	**F_1**	**F_2 to M_3**
After cleaning with alcohol	1 to 2 days	1 hour
After cleaning with distilled water	1 day	1 hour

2.5.1.4. Prior to performing any calibration tests, the weights need to be acclimated to the ambient conditions of the laboratory. In particular, weights of classes F1 (or better) should be close to the temperature in the weighing area and equilibrate for a minimum of 24 hours. The minimum times (in hours) required for temperature stabilization (depending on weight size, weight class and on the difference between the initial temperature of the weights and the room temperature in the laboratory) are shown in the table below (with appropriate documented evidence). As a practical guideline, a waiting time of 24 hours is recommended. If weights are extremely hot or frozen additional equilibration may be needed to

address problems with condensation and frozen surfaces. Weights must be completely dry prior to calibration.

Table 4. Minimum equilibration times[5].

ΔT*	Nominal Mass	OIML Class F_1 (time in h)	OIML Class F_2 to M_3 (time in h)
± 20 °C	1 000, 2 000, 5 000 kg	79	5
	100, 200, 500 kg	33	4
	10, 20, 50 kg	12	3
	1, 2, 5 kg	6	2
	100, 200, 500 g	3	1
	10, 20, 50 g	1	1
	< 10 g	1	0.5
± 5 °C	1 000, 2 000, 5 000 kg	4	1
	100, 200, 500 kg	4	1
	10, 20, 50 kg	4	1
	1, 2, 5 kg	3	1
	100, 200, 500 g	2	1
	< 100 g	1	1
± 2 °C	<100 g to 5 000 kg	1	0.5
± 0.5 °C	<100 g to 5 000 kg	0.5	0.5

*ΔT = Initial difference between weight temperature and laboratory temperature.

2.5.1.5. Conduct preliminary measurements to obtain an approximate value for the difference between the standard and the unknown, to identify where in the weighing instrument range the readings occur, to determine if tare weights are required, to determine the sensitivity weight to be used, and to determine the time interval

[5] Consider equivalent ASTM Classes for equilibration times.

required for the weighing instrument indication to stabilize. See NISTIR 6969, SOP 34 for specific instructions on evaluation of the need for and selection of tare weights and sensitivity weights.

Tare weights are rarely needed for mass standards that are within applicable tolerances. When unequal nominal weights are compared, tare weights are often required. When tare weights are required, carry tare weights, t_s and t_x, with the standard and the unknown, S and X, respectively. The tare weights must be calibrated standards with valid uncertainties that are included in the process of determining mass values and calibration uncertainties. The standard and its tare weight, $S + t_s$, should be "nearly the same mass" as the unknown with its tare weight, $X + t_x$. "Nearly the same mass" depends upon the balance used (See SOP 34, Table 1).

A sensitivity weight must be used on equal-arm balances, single-pan mechanical, and is usually used on electronic balances, to ensure that the measured differences between the standard(s) and unknown test items have valid accuracy and traceability (See SOP 34, Table 2) (e.g., the optical/digital scale is *calibrated* each time the procedure is performed through the use of a sensitivity weight).

If the sensitivity of the balance has been analyzed and is periodically evaluated and found to introduce negligible errors or uncertainties according to SOP 34 and measurement requirements, the equations in Section 3 must be modified to eliminate the sensitivity factor to avoid “division by zero” errors in the calculations.

If a sensitivity weight will be used, select one that meets the criteria in SOP 34.

2.5.1.6. Determine whether optional sequence A or B will be used. Optional sequence A uses the standard on the balance for the first observation and the unknown on the balance for the second and third observations; this is often called the “*SXX*” sequence. Optional sequence B starts with the unknown on the balance first and with the standard on the balance for the second and third observations; this is often called the “*XSS*” sequence.

2.5.1.7. Adjust the single pan balance or the combination balance so the first two readings of the single substitution fall in the first quarter of the optical scale or digital indications. The zero

adjustment and tare adjustment may be used. Small weights may be placed on the balance pan to reach the desired reading range. These weights remain on the pan throughout the single substitution. Once the balance has been adjusted to the desired position, neither the balance dials, the zero and tare adjustments, nor the small weights placed on the balance pan are to be changed during the measurement.

2.5.1.8. If the balance is equipped with a pan arrestment mechanism, arrest the pan between each observation.

2.5.2. Measurement Procedure, Optional Sequence A (*SXX*)

Table 5. Optional Sequence A.

Measurement No.	Weights on Pan	Observation
1	$S + t_s$	O_1
2	$X + t_x$	O_2
3	$X + t_x + sw$	O_3

All observations should be recorded on suitable data sheets, such as those in the appendix. Record the laboratory ambient temperature, barometric pressure, and relative humidity.

2.5.1.1. Observation 1. Place the standard weight(s), *S*, along with t_s on the balance pan. If equipped with a pan arrestment mechanism, release the balance pan. When the pan is released, start the stop-watch and record observation O_1 once the balance indication has stabilized.

2.5.1.2. Observation 2. Remove weight(s) *S* and t_s and replace with test weight *X* and its tare weight, t_x. Release the pan, time the interval, and record observation O_2.

2.5.1.3. Observation 3. Add the sensitivity weight, *sw*, to the weights of observation 2. Release the pan, time the interval, and record observation O_3.

2.5.1.4. If repeated single substitutions are performed, the values between successive trials should not differ from one another by more than ± 2 sd of the balance. If this difference is exceeded, reject the data and take a new series of measurements that agree.

2.5.3. Measurement Procedure, Optional Sequence B (*XSS*)

Measurements for Option B are made as described in Option A except that X, S, t_x, and t_s are interchanged.

Table 6. Optional Sequence B.

Measurement No.	Weights on Pan	Observation
1	$X + t_x$	O_1
2	$S + t_s$	O_2
3	$S + t_s + sw$	O_3

3. Calculations

3.1. If no air buoyancy correction is performed, calculate the conventional mass correction, C_x, for the test weight as follows, according to the optional sequence used. Incorporate an uncorrected systematic uncertainty for the magnitude of the buoyancy correction in the uncertainty calculations according to SOP 2 if using this equation. In each case, the conventional mass corrections for the standard weight(s), C_s, the conventional mass of the tare weights, CM_{t_s} and CM_{t_x}, and the conventional mass of the sensitivity weight, CM_{sw}, are included. The symbols N_s and N_x refer to the nominal values of S and X, respectively. If nominal weights are equal and no tare weights are used, values for nominal values and tare weights may be entered as zero.

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3.1.1. Optional Sequence A (*SXX*)

$$C_x = C_s + CM_{t_s} - CM_{t_x} + (O_2 - O_1)\left[\frac{CM_{sw}}{(O_3 - O_2)}\right] + N_s - N_x$$

3.1.2. Optional Sequence B (*XSS*)

$$C_x = C_s + CM_{t_s} - CM_{t_x} + (O_1 - O_2)\left[\frac{CM_{sw}}{(O_3 - O_2)}\right] + N_s - N_x$$

3.2. Mass Calculation with Air Buoyancy Correction

3.2.1. Calculate the air density, ρ_a, as described in the Appendix to SOP No. 2.

3.2.2. Calculate the mass of the test weight, M_x, and its mass correction C_x using the mass of the standard weight(s), the tare weights and the sensitivity weights according to the optional sequence used.

3.2.2.1. Optional Sequence A (*SXX*)

$$M_x = \frac{M_s\left(1 - \frac{\rho_a}{\rho_s}\right) + M_{t_s}\left(1 - \frac{\rho_a}{\rho_{t_s}}\right) - M_{t_x}\left(1 - \frac{\rho_a}{\rho_{t_x}}\right) + (O_2 - O_1)\left[\frac{M_{sw}\left(1 - \frac{\rho_a}{\rho_{sw}}\right)}{(O_3 - O_2)}\right]}{\left(1 - \frac{\rho_a}{\rho_x}\right)}$$

3.2.2.2. Optional Sequence B (*XSS*)

$$M_x = \frac{M_s\left(1 - \frac{\rho_a}{\rho_s}\right) + M_{t_s}\left(1 - \frac{\rho_a}{\rho_{t_s}}\right) - M_{t_x}\left(1 - \frac{\rho_a}{\rho_{t_x}}\right) + (O_1 - O_2)\left[\frac{M_{sw}\left(1 - \frac{\rho_a}{\rho_{sw}}\right)}{(O_3 - O_2)}\right]}{\left(1 - \frac{\rho_a}{\rho_x}\right)}$$

3.2.3. Calculate the mass correction C_x, as follows:

$$C_x = M_x - N_x$$

where N_x is the nominal value for X.

3.2.4. Calculate the conventional mass[6] of X, CM_x. It is recommended that the conventional mass be reported.

3.2.4.1. Conventional mass

[6] Conventional Mass: "The conventional value of the result of weighing a body in air is equal to the mass of a standard, of conventionally chosen density, at a conventionally chosen temperature, which balances this body at this reference temperature in air of conventionally chosen density." The conventions are: reference density 8.0 g/cm^3; reference temperature 20 °C; *normal* air density 0.0012 g/cm3. Conventional mass was formerly called "Apparent Mass versus 8.0 g/cm^3" in the United States. *See OIML D28 (2004).*

$$CM_x = \frac{M_x \left(1 - \frac{\rho_n}{\rho_x}\right)}{\left(1 - \frac{\rho_n}{8.0}\right)}$$

3.2.5. If requested, the apparent mass versus the reference density of brass may be calculated. This value should only be provided when requested by the customer for use when calibrating mechanical balances that have been adjusted to this reference density. (This is rare).

3.2.5.1. Apparent mass versus brass

$$\mathrm{AM}_{\mathrm{x\ vs\ brass}} = \frac{\mathrm{M_x} \left(1 - \frac{\rho_\mathrm{n}}{\rho_\mathrm{x}}\right)}{\left(1 - \frac{\rho_\mathrm{n}}{8.3909}\right)}$$

4. Measurement Assurance

4.1. Duplicate the process with a suitable check standard (See GLP 1, SOP 9, SOP 30, and Sec. 7.4).

4.2. Plot the check standard value and verify that it is within established limits; a t-test may be incorporated to check observed value against accepted value.

4.3. The mean value of the check standard is compared to a reference value of the check standard with respect to their applicable expanded uncertainties to evaluate bias and drift over time.

4.4. Check standard observations are used to calculate the standard deviation of the measurement process, s_p.

4.5. Where SOP 9 is followed and check standards are used for 2 to 3 nominal values per balance, more frequent calibration intervals may be needed to monitor the working standards and the larger standard deviation of the nominal values bracketing the nominal value should be used. If check standards were already checked on a given day and found to be in control, additional evaluations may be conducted, but are not required.

5. Assignment of Uncertainty

The limits of expanded uncertainty, U, include estimates of the standard uncertainty of the mass standards used, u_s, estimates of the standard deviation of the measurement process, s_p, and estimates of the effect of other components associated with this procedure, u_o. These estimates should be combined using the root-sum-squared method

(RSS), and the expanded uncertainty, *U*, reported with a coverage factor of two (k=2), to give us an approximate 95 % level of confidence. See SOP 29 for the complete standard operating procedure for calculating the uncertainty.

5.1. The expanded uncertainty for the standard, *U*, is obtained from the calibration certificate. The combined standard uncertainty, u_c, is used and not the expanded uncertainty, *U*, therefore the reported uncertainty for the standard will usually need to be divided by the coverage factor k.

5.2. The value for s_p is obtained from the control chart data for check standards using single substitution measurements. (See SOP No. 9.)

5.3. Include an uncorrected systematic standard uncertainty if no buoyancy correction was performed. Calculate the magnitude of the air buoyancy correction per SOP 2 and use a rectangular distribution.

5.4. Other standard uncertainties that may be included at this calibration level include uncertainties associated with calculation of air density if buoyancy corrections are performed, standard uncertainties associated with the density of the standards used, and any noted bias that has been determined through analysis of control charts and round robin data. See SOP 2, 4, and 29 for additional details.

5.5. Example components to be considered for an uncertainty budget table are shown in the following table.

Table 7. Example uncertainty budget table.

Uncertainty Component Description	Symbol	Source	Typical Distribution
Uncertainty of the standard mass(es) (5.1)	u_s	Calibration certificate	Rectangular or Normal divided by coverage factor
Accepted standard deviation of the process (5.2)	s_p	Control chart, standard deviation chart	Normal
Uncertainty of the uncorrected air buoyancy correction (5.3)	u_b	SOP 2	Rectangular
Uncertainty associated with bias (5.4)	u_d	Control chart, proficiency tests	See SOP 29

5.6. Evaluate compliance to applicable tolerances as needed by the customer. The expanded uncertainty, U, must be $\leq 1/3$ of the applicable tolerances published in ASTM E617 and OIML R111 standards. Additionally, the mass value plus the expanded uncertainty must be less than the applicable tolerance to confidently state that mass standards are in or out of tolerance.

5.7. Draft a suitable uncertainty statement for the certificate.

For example: The uncertainty reported is the root sum square of the standard uncertainty of the standard, the standard deviation of the process, and an uncorrected systematic error for lack of buoyancy corrections, multiplied by a coverage factor of 2 ($k = 2$) for an approximate 95 percent confidence interval. Factors not considered in the evaluation: magnetism (weights are considered to meet magnetism specifications unless measurement aberrations are noted), balance eccentricity and linearity (these factors are considered as a part of the measurement process when obtaining the standard deviation of the process when using a check standard with adequate degrees of freedom.)

6. Certificate

Report results as described in SOP No. 1, Preparation of Calibration Certificates.

Appendix
Single Substitution Data Sheet
(Optional Sequence A, *SXX)*

Laboratory data and conditions:

			Before	After
Operator			Before	After
Date		Temperature		
Balance		Pressure		
Load		Relative Humidity		
Standard deviation of the process, from control chart, s_p		Degrees of Freedom		

Mass standard(s) data:

ID	Nominal	Mass Correction	Unc: From cal. certificate	Unc: k factor	Density g/cm^3
S					
t_s					
X					
t_x					
S_c					
t_{Sc}					
sw					

Mass Correction = *True Mass* if using buoyancy correction. Mass Correction = *Conventional Mass* if NOT using buoyancy correction. Density is used only with buoyancy corrections.

Observations:

Measurement #	Weights	Balance Observations, Units _____
Time:		
1 (O_1)	$S + t_s$	
2 (O_2)	$X + t_x$	
3 (O_3)	$X + t_x + sw$	
Time:		

Measurement Assurance (Duplication of the Process):

Measurement #	Weights	Balance Observations, Units _____
Time:		
1 (O_1)	$S + t_s$	
2 (O_2)	$S_c + t_{Sc}$	
3 (O_3)	$S_c + t_{Sc} + sw$	
Time:		
Check Standard in Control?	_____Yes_____No	

Appendix
Single Substitution Data Sheet
(Optional Sequence B, *XSS)*

Laboratory data and conditions:

			Before	After
Operator				
Date		Temperature		
Balance		Pressure		
Load		Relative Humidity		
Standard deviation of the process, from control chart, s_p		Degrees of Freedom		

Mass standard(s) data:

ID	Nominal	Mass Correction	Unc: From cal. certificate	Unc: k factor	Density g/cm^3
S					
t_s					
X					
t_x					
S_c					
t_{Sc}					
sw					

Mass Correction = *True Mass* if using buoyancy correction. Mass Correction = *Conventional Mass* if NOT using buoyancy correction. Density is used only with buoyancy corrections.

Observations:

Measurement #	Weights	Balance Observations, Units _____
Time:		
1 (O_1)	$X + t_x$	
2 (O_2)	$S + t_s$	
3 (O_3)	$S + t_S + sw$	
Time:		

Measurement Assurance (Duplication of the Process):

Measurement #	Weights	Balance Observations, Units _____
Time:		
1 (O_1)	$S + t_s$	
2 (O_2)	$S_c + t_{Sc}$	
3 (O_3)	$S_c + t_{Sc} + sw$	
Time:		
Check Standard in Control?	_____Yes_____No	

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SOP No. 8

Recommended Standard Operating Procedure for Medium Accuracy Calibration of Mass Standards by Modified Substitution

1. Introduction

1.1. This SOP describes procedures to be followed for determining whether or not mass standards are within the tolerances specified for a particular class of standards (e.g., NIST Class F, ASTM[1] Class 5, 6, 7 or OIML[2] Classes M_1, M_2, and M_3) where the uncertainty is usually much smaller than the tolerance application provided the laboratory maintains standards and balances used for higher precision work. This procedure permits the metrologist to report that the weights under test were compared against a reference standard with the results reported on the laboratory calibration certificate. The comparison is important because the built-in weights of a balance do not represent laboratory standards or provide metrological traceability unless they have been formally calibrated. If a State law or other regulation requires that field weights be compared against the State (or reference) standards, this procedure can be used to fulfill this requirement. It is suitable for calibration when moderate accuracy is required, and does not eliminate errors due to drift. The procedure does not incorporate measurement control steps to ensure the validity of the standards and the measurement process; therefore, additional precautions are described for the use of check standards. The expanded uncertainty with this procedure must be ≤ 1/3 of the tolerance per ASTM and OIML standards. If uncertainties to tolerance ratios are greater than required, SOP 7 (single substitution) or SOP 4 (double substitution) procedures are preferred.

Note: If you use SOP 8, you are most likely using working standards that are equivalent or only slightly better than your customer's weights. If this is the case, the working standards must have a higher level of calibration than the weights being calibrated. Therefore, you will need standards, balances, procedures and uncertainties better than the customer weights and this procedure to calibrate your own working standards.

1.2. Prerequisites

1.2.1. Valid calibration certificates with appropriate values and uncertainties must be available for all of the standards used in the calibration. All standards must have demonstrated metrological traceability to the

[1] ASTM International (formerly the American Society for Testing and Materials) publishes the E617 standard for mass specifications and tolerances.

[2] OIML is the International Organization for Legal Metrology. Weight classes are published in OIML R 111, which is freely available at http://www.oiml.org.

international system of units (SI), which may be to the SI, through a National Metrology Institute such as NIST.

1.2.2. Standards must be evaluated to ensure that standard uncertainties for the intended level of calibration are sufficiently small. Reference standards should not be used to routinely calibrate customer standards using this procedure.

1.2.3. Verify that the balance that is used is in good operating condition with sufficiently small process standard deviation as verified by a valid control chart or preliminary experiments to ascertain the performance quality when a new balance is put into service.

1.2.4. Verify that the operator is experienced in precision weighing techniques and has had specific training in SOP 7, SOP 8, SOP 29, and GMP 10.

1.2.5. Laboratory facilities must comply with the following minimum conditions to meet the expected uncertainty possible with this procedure and to comply with the balance manufacturer's operating conditions specified for the balance. The laboratory must have demonstrated environmental controls for a minimum of 24 hours before a calibration.

Table 1. Environmental conditions.

Echelon[3]	Temperature Requirements During a Calibration	Relative Humidity (%)
III	Lower and upper limits: 18 °C to 27 °C Maximum changes: ± 5 °C / 12 h and ± 3 °C / h	40 to 60 ± 20 / 4 h

1.2.5.1. As a practical guideline, a waiting time of 24 hours is recommended for weights calibrated with this procedure to become equilibrated in the laboratory. If weights are extremely hot or cold additional equilibration may be needed to address problems with condensation and frozen surfaces. Weights must be completely dry prior to calibration. Minimum equilibration times are provided in the following table.

Table 2. Stabilization time.

Temperature Range for the Artifact	Minimum Equilibration Time
Inside upper or lower limits < 18 ºC or > 27 ºC	None
Outside of upper or lower limits < 18 ºC or > 27 ºC	24 hours/overnight

2. Methodology

[3] Echelon III corresponds to weights of Classes OIML M_1, M_2, and M_3 or NIST Class F. Uncertainty values must be fully assessed if this procedure is attempted for any higher class weights. This procedure does not provide adequate redundancy for OIML Classes E_1 or E_2 and SOP 5 or SOP 28 must be used.

2.1. Scope, Precision, Accuracy

This method is applicable to all lower echelon mass calibration (tolerance testing) provided that the uncertainty requirements can be met. The achievable precision using this procedure is appropriate, provided the expanded uncertainty of the measurement is no more than one-third of the permissible tolerance of the mass standard tested. The accuracy achievable with this procedure depends on the accuracy of the calibration of the working standards and the precision of the intercomparison.

2.2. Summary

The mass to be tested is compared with a calibrated working standard by a modified substitution procedure. The comparison may be made using a single-pan, an equal-arm, or a fully-electronic balance. The reference standard is placed on the balance to obtain a convenient reference point and a sensitivity test is conducted. The error (departure from nominal value) of the weight tested is determined by comparing its reading to the reading obtained for the reference standard. A weight is considered to be within tolerance when the absolute value of its error plus its uncertainty does not exceed the tolerance established for the particular class of weight.

2.3. Apparatus/Equipment

2.3.1. Single-pan (Option A and A_1), full-electronic balance (Option B), or equal-arm balance (Option C) with sufficient capacity for the load tested and with resolution equal to or less than one-tenth of the applicable tolerance of the standards to be tested.

2.3.2. Calibrated working standards, of nominally equal mass to the unknown mass standards being calibrated. Mass standards must have an expanded uncertainty less than one-tenth of the tolerance tested.

2.3.3. Calibrated sensitivity weights, and tare weights if needed, with current calibration certificates selected according to SOP 34.

2.3.4. Uncalibrated counterweights, *T*, of approximately the same mass as the standard weights (for option C).

2.3.5. Calibrated environmental equipment to monitor laboratory conditions and to perform buoyancy corrections if needed. See SOP 2 for appropriate accuracy limits.

2.4. Procedure - Option A, Use of Single-Pan Mechanical Balance

2.4.1. Select a reference standard of the same nominal value as the weight under test. Place the standard on the balance pan. Adjust the optical scale reading (See GMP No. 4) to approximately midscale using uncalibrated tare

weights and the balance's coarse and fine Zero controls. This setting must not be altered during a measurement sequence. Record the reading as O_1.

2.4.2. Add a sensitivity weight equal to approximately one-quarter full scale reading and record reading as O_2.

2.4.3. Calculate the value of a scale division using the equation in 3.2. If it is within ± 2 % of nominal value (usual case) the nominal value of a division can be used for tolerance testing.

2.4.4. Remove the sensitivity weight and adjust the optical scale to account for corrected value of standard used.

Example: Suppose that the nominal range of the optical scale is 100 mg and that the reference standard has a correction of -2.5 mg. The optical scale is adjusted to read 47.5 mg when the standard is on the pan. Under this condition, the reading 50.0 mg represents the nominal mass of the reference standard.

2.4.5. Remove the Standard.

2.4.6. Place the weight to be tested on the balance pan, read the optical scale and record reading as X_n. The error in the weight is the amount by which the indication deviates from the mid-scale reading. If the weight indication is more than the mid-scale value, the weight is heavy by the indicated difference; if the indication is less than the mid-scale value, the weight is light.

2.4.7. After several weights have been tested ((no more than 10 unknown weights may be tested without rechecking the standard or check standard), put the standard on the balance pan and record the reading. The difference between this indication and the previous one for the standard indicates a balance drift. This drift will normally be very small. If the drift exceeds 10 % of the tolerance applicable to the weights under test or affects a measurement result to the extent that a weight may be out of tolerance, the measurement should be repeated and more frequent checks of the standard should be made or a more appropriate procedure should be used. The average drift may be monitored over time to be included as a component of the uncertainty; otherwise 10 % of the applicable tolerance, as a rectangular distribution may be included in the uncertainty calculations.

2.4.8. Readjust the optical scale at any time that a significant difference is observed when rechecking a standard.

2.4.9. Calculate the mass correction for the unknown weights using the appropriate equation in Section 3.

2.5. Procedure - Option A_1 Use of Single-Pan Mechanical Balance

2.5.1. Select a reference standard of the same nominal value as the weight under test. Place the standard on the balance pan. Adjust the optical scale reading (See GMP No. 4) to midscale using uncalibrated tare weights and the balance's coarse and fine Zero controls. This setting must not be altered during a measurement sequence. Record the reading as O_1.

2.5.2. Add a sensitivity weight equal to approximately one-quarter full scale reading and record reading as O_2.

2.5.3. Calculate the value of a scale division using the equation in 3.2. If the sensitivity is within ± 2 % of nominal value (usual case) of the scale division, the nominal value of a division may be used.

2.5.4. Remove the sensitivity weight and re-adjust the optical scale to obtain a midscale indication, if the indication has changed from that set in 2.5.1.

2.5.5. Remove the Standard.

2.5.6. Place the weight to be tested on the balance pan, read the optical scale and record the indication as X_n. The error in the weight is the amount by which the indication deviates from the mid-scale reading. If the weight indication is more than the mid-scale value, the weight is heavier than the standard by the indicated difference; if the indication is less than the mid-scale value, the weight is lighter than the standard.

2.5.7. After several weights have been tested (no more than 10 unknown weights may be tested without rechecking the standard or check standard) put the standard on the balance pan and record the reading. The difference between this indication and the previous one for the standard indicates a balance drift. This drift will normally be very small. If the drift exceeds 10 % of the tolerance applicable to the weights under test or affects a measurement result to the extent that a weight may be out of tolerance, the measurement should be repeated and more frequent checks of the standard should be made or a more appropriate procedure should be used. The average drift may be monitored over time to be included as a component of the uncertainty; otherwise 10 % of the applicable tolerance, as a rectangular distribution may be included in the uncertainty calculations.

2.5.8. Readjust the optical scale at any time that a significant difference is observed when rechecking a standard.

2.5.9. Calculate the correction of the unknown using the equation

$$C_x = C_s + (X_n - O_1)$$

2.6. Procedure - Option B, Use of Full Electronic Balance

2.6.1. Select a reference standard of the same nominal value as the weight under test. Place the standard on the pan. Zero the balance and record the stable reading as O_1. A stopwatch may be used to monitor consistent timing of balance stability but is not required.

2.6.2. Add a calibrated sensitivity weight (sw ≥ 2 times the tolerance but not exceeding 0.5 % of the balance capacity) and record the reading as O_2. Verify whether the nominal scale division is within ± 2 % of nominal value of the scale division using the equation in 3.2. If so, the nominal value of the scale division may be used.

2.6.3. Remove sensitivity weight and zero the balance so weight differences, *d*, can be read directly from the balance indications.

2.6.4. Remove all weights from the balance pan.

2.6.5. Place the weight to be tested on the balance pan. Record the reading as X_n.

2.6.6. After several weights have been tested (no more than 10 unknown weights may be tested without rechecking the standard or check standard) recheck the zero and record the reading. The difference between this indication and the previous one for the standard indicates a balance drift. This drift will normally be small. If the drift exceeds 10 % of the tolerance applicable to the weights under test or affects a measurement result to the extent that a weight may be out of tolerance, the measurement should be repeated and more frequent checks of the standard should be made or a more appropriate procedure should be used. The average drift may be monitored over time to be included as a component of the uncertainty; otherwise 10 % of the applicable tolerance, as a rectangular distribution may be included in the uncertainty calculations.

2.6.7. Readjust the zero at any time that a significant difference is observed when rechecking a standard.

2.6.8. Calculate the mass correction for each weight using the equation

$$C_x = C_s + (X_n - O_1) = C_s + X_n \text{ (when } O_1 \text{ is zeroed)}$$

2.7.Procedure - Option C, Use of Equal Arm Balance

2.7.1. Select a reference standard of the same nominal value as the weight under test. Place the standard on the left balance pan together with small, calibrated weights equal to the correction required for the standard, provided it is light. If needed, add sufficient counterweights to the right pan to obtain a sum of turning points of approximately twice midscale value. If necessary, number the graduated scale such that adding weights

to the left pan will increase the balance reading. Record the sum of the turning points as 0_1.

2.7.2. Add an appropriate calibrated sensitivity weight to the left pan and record the sum of the turning points as 0_2. Calculate the sensitivity,

$$sensitivity = \frac{CM_{sw}}{(O_2 - O_1)}$$

where CM_{sw} is the conventional mass of the sensitivity weight.

2.7.3. Remove all weights from the left pan.

2.7.4. Place weight to be tested on the left pan. If the standard used in 2.7.1 was heavy, add small correction weights to the left pan, equivalent to the correction required for the standard. Add small, calibrated tare weights as required to left or right pan to obtain an approximate balance and record the sum of the turning points as X_n.

2.7.5. Calculate the mass correction of the weight tested as follows.

2.7.5.1. If added tare weights are placed on the left pan (t_x).

$$C_x = (X_n - O_1)\left[\frac{CM_{sw}}{(O_2 - O_1)}\right] - CM_{t_x}$$

2.7.5.2. If added tare weights are placed on the right pan (t_s)

$$C_x = (X_n - O_1)\left[\frac{CM_{sw}}{(O_2 - O_1)}\right] + CM_{t_s}$$

2.7.6. After several weights have been tested (no more than 10 unknown weights may be tested without rechecking the standard or check standard), recheck the turning point 0_1, as described in 2.6.1. Only a small difference should be observed. If the difference exceeds 25 % of the sum of the turning points in O_1, the measurement should be repeated and more frequent checks of the standard should be made or a more appropriate procedure should be used. The average drift may be monitored over time to be included as a component of the uncertainty; otherwise 10 % of the applicable tolerance, as a rectangular distribution may be included in the uncertainty calculations.

2.8.Tolerance Evaluation

2.8.1. Compare the correction plus the expanded uncertainty of the weight tested with the tolerance for the class of weights to which it belongs. If the absolute value of the correction plus the expanded uncertainty is numerically smaller than the tolerance, the weight is considered to be within tolerance. If the correction is larger than permissible, the weight is considered to be outside the tolerance and appropriate action should be taken. It is recommended that weights whose absolute value of the correction exceeds 75 % of the tolerance limit be adjusted closer to the nominal value where possible. Laboratories may set suitable alternative limits, taking care to ensure that the mass value plus (or minus) the uncertainty is within the tolerance limits.

Table 3. Example of weighing sequence.

Measurement No.	Weights on Pan	Observation
1	S	O_1
2	$S + sw$	O_2
3	S	O_3
4	X_1	O_4
5	X_2	O_5
6	X_3	O_6
7	X_4	O_7
8	X_5	O_8
9	X_6	O_9
10	X_7	O_{10}
11	X_8	O_{11}
12	X_9	O_{12}
13	X_{10}	O_{13}
14	S_c	O_{14}
15	S	O_{15}

3. Calculations

3.1. Air buoyancy corrections are generally *not* made with the modified substitution, although with the use of spreadsheets and the need to record environmental conditions, there is no reason why the buoyancy correction may not be routinely included (mass densities must be known or assumed). Calculate the conventional mass correction, C_x, for the test weight as follows, according to the optional sequence used. In each case, the conventional mass corrections for the standard weight(s) are included.

Table 4. Symbols used.

Symbol	Description
CM_i	conventional mass of weight i
N_s	nominal value of S
N_x	nominal value of X

3.2. Evaluate the sensitivity of the balance:

$$sensitivity = \frac{CM_{sw}}{(O_2 - O_1)}$$

If the sensitivity error is less than 2 % of the nominal value of a division on the optical scale or the electronic range of operation, proceed with the modified substitution. If the sensitivity error is greater, SOP 7, Single Substitution may be acceptable. If corrections for sensitivity are not made, an uncorrected systematic error may be incorporated into the uncertainty but the balance sensitivity requires periodic evaluation; this value may be calculated as 2 % of the applicable tolerance.

3.3. Calculate the mass correction of each unknown weight as follows if the correction for the standard IS NOT used in setting a reference point on the balance:

$$C_x = C_s + d$$

$$C_x = C_s + (X_n - O_1)$$

Note: If an electronic balance is used and zeroed with the standard on the balance, O_1 is "0" and d becomes the X_n balance reading.

3.4. Calculate the mass correction of each unknown weight as follows if the correction for the standard IS used in setting a nominal reference point on the balance:

$$C_x = (X_{balance\ reading} - N_{nominal\ reference\ point})$$

Note: In this case the standard and its correction are used to artificially *set* a perfect nominal reference point for use in comparing the unknown weights.

3.5. If tare weights and unequal nominal values are used, use the following equation for modifying section 3.3:

$$C_x = C_s + CM_{ts} - CM_{tx} + (X_n - O_1) + N_s - N_x$$

4. Measurement Assurance

4.1. Duplicate the process with suitable check standards for each nominal load or a designated number of check standards per balance (See GLP 1, SOP 9, SOP 30, and Sec. 7.4).

4.2. Evaluate the value against the expected limits and plot the check standard value to monitor changes over time; a t-test may be incorporated to check the observed value against the accepted value.

4.3. The mean value of the check standard is compared to a reference value of the check standard with respect to their applicable expanded uncertainties to evaluate bias and drift over time.

4.4. Check standard observations are used to calculate the standard deviation of the measurement process, s_p.

4.5. Where SOP 9 is followed and check standards are used for 2 to 3 nominal values per balance, more frequent calibration intervals may be needed to monitor the working standards and the larger standard deviation of the nominal values bracketing the nominal value should be used. If check standards were already checked on a given day and found to be in control, additional evaluations may be conducted, but are not required.

5. Assignment of Uncertainty

The limits of expanded uncertainty, U, include estimates of the standard uncertainty of the mass standards used, u_s, estimates of the standard deviation of the measurement process, s_p, and estimates of the effect of other components associated with this procedure, u_o. These estimates should be combined using the root-sum-squared method (RSS), and the expanded uncertainty, U, reported with a coverage factor of two (k=2), to give an approximate 95 % level of confidence. See SOP 29 for the complete standard operating procedure for calculating the uncertainty.

5.1. The expanded uncertainty for the standard, U, is obtained from the calibration certificate. The combined standard uncertainty, u_c, is used and not the expanded uncertainty, U, therefore the reported uncertainty for the standard will usually need to be divided by the coverage factor k.

5.2. Standard deviation of the measurement process from control chart performance (See SOP No. 9.) The value for s_p is obtained from the control chart data for check standards using modified substitution measurements.

5.3. Uncertainty associated with allowable drift. Include the monitored average drift for each balance and procedure as a rectangular distribution, or include a value that is approximately 10 % of the applicable tolerance for the weight, again as a rectangular distribution. Whenever the drift exceeds 10 %, conduct a root cause analysis to determine the cause; this situation may require fewer weights to be calibrated in the weighing series, service of the balance, better environmental controls, or additional training and practice by the metrologist.

5.4. Uncertainty associated with allowable sensitivity error. This procedure allows for up to 2 % error in the optical or electronic range of use. This may result in calibration errors up to 2 % of the applicable tolerance. This is potentially another uncorrected systematic error that may be treated as a rectangular distribution.

5.5. Uncertainty associated with uncorrected air buoyancy. When buoyancy corrections are not made, the resulting uncorrected systematic error may be treated as a rectangular distribution, with the magnitude of the maximum

uncorrected buoyancy correction determined using equations provided in SOP 2. The uncertainty associated with air density and its associated factors may be considered negligible for this procedure or may be calculated using the formula provided in SOP 4 or 5.

5.6. Uncertainty associated with bias that is observed in the check standards as determined through analysis of the control charts. When bias is observed in the control charts, it must be assessed according to the equations provided in SOP 2 and may be incorporated as an uncorrected systematic error using the equations in SOP 29.

5.7. Example components to be considered for an uncertainty budget table are shown in the following table.

Table 5. Example uncertainty budget table.

Uncertainty Component Description	Symbol	Source	Typical Distribution
Uncertainty of the standard mass(es) (5.1)	u_s	Calibration certificate	Rectangular or Normal divided by coverage factor
Accepted standard deviation of the process (5.2)	s_p	Control chart, standard deviation chart	Normal
Uncertainty of the allowable drift (5.3)	u_{drift}	10 % of the tolerance	Rectangular
Uncertainty of the allowable sensitivity error (5.4)	u_{sw}	SOP 8, 2 % of the sensitivity	Rectangular
Uncertainty of the maximum magnitude of the buoyancy correction (5.5)	u_b	SOP 2	Rectangular
Uncertainty associated with bias (5.6)	u_d	Control chart, proficiency tests	See SOP 29

5.8. Evaluate the expanded uncertainty, which must be less than 1/3 of the tolerance applicable as per ASTM E617, OIML R111, or NIST Class F (via Handbook 44, Fundamental Considerations).

5.9. Draft a suitable uncertainty statement for the certificate.

For example: The uncertainty reported is the root sum square of the standard uncertainty of the standard, the standard deviation of the process, and an uncorrected systematic error for lack of buoyancy corrections, multiplied by a coverage factor of 2 ($k = 2$) for an approximate 95 percent confidence interval. Factors not considered in the evaluation: magnetism (weights are considered to meet magnetism specifications unless measurement aberrations are noted), balance eccentricity and linearity (these factors are considered as a part of the measurement process when obtaining the standard deviation of the process when using a check standard with adequate degrees of freedom.)

6. Certificate

Report results as described in SOP No. 1, Preparation of Calibration Certificates.

Appendix - Modified Substitution Data Sheet

Laboratory data and conditions:

			Before	After
Operator				
Date		Temperature		
Balance		Pressure		
Load		Relative Humidity		
Standard deviation of the process, from control chart, s_p		Degrees of Freedom		

Mass standard(s) data:

ID	Nominal	Mass Correction*	Expanded Unc: from cal. certificate	Unc: k factor	Density g/cm^3
S					
t_S					
X		TBD	TBD	TBD	
t_x					
S_c					
t_{Sc}					
sw					

*Mass Correction = *True Mass* with buoyancy correction. Mass Correction = *Conventional Mass* with no buoyancy correction.

Observations:

Observation No.	Weights (Insert ID of X)	Balance Observations, Units
Time:		
1 (O_1)	S	
2 (O_2)	$S + sw$	
	Error < 2 % of optical scale or electronic range? _____Yes_____No	
3 (O_3)	S	
4 (O_4)	X_1	
5 (O_5)	X_2	
6 (O_6)	X_3	
7 (O_7)	X_4	
8 (O_8)	X_5	
9 (O_9)	X_6	
10 (O_{10})	X_7	
11 (O_{11})	X_8	
12 (O_{12})	X_9	
13 (O_{13})	X_{10}	
14 (O_{14})	S_c	
15 (O_{15})	S	
Time:	Drift < 1/10 Tolerance ?: _____Yes_____No	
Check Standard in Control?	_____Yes_____No	

Up to 10 unknown weights may be checked with this procedure if the drift is less than 1/10 of the tolerance.

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SOP No. 9

Recommended Standard Operating Procedure for Control Charts for Calibration of Mass Standards

1. Introduction

1.1. This SOP describes procedures for the development of control charts and their use for demonstrating attainment of statistical control of a mass calibration process. The procedure may be applied to other calibration processes as well. See also NISTIR 6969, SOP 30 and NISTIR 7383, SOP 17 and 20.

1.2. Prerequisites

The use of this SOP requires that appropriate apparatus, methodology, and standards are available, and that the laboratory thoroughly understands the basic principles of the measurement process used and has had sufficient experience to perform the necessary operations required for the measurements of concern.

2. Summary

An appropriate check standard (or control standard) is incorporated into the measurement process and weighed at established intervals; the results are plotted on an $\bar{x}$ (X-bar) chart. The abscissa (x) represents the sequence of measurements and the ordinate (y) the measured values. A central line is drawn, indicating the mean ($\bar{x}$) of the measured values and control limits are indicated within which the results of measurements are expected to be randomly distributed, based on statistical considerations. The system is considered to be in statistical control when the individual values are within the designated limits. The system is considered to be out of control if values are present outside established limits for which no reasonable and correctable cause have been determined and corrected, unusual trends are observed, or if the mean exceeds the control limits. The statistical information on which the control limits are based can be used to calculate confidence limits for measurements made while the system is demonstrated to be stable and in a state of statistical control.

3. Procedure

3.1. Definition of Monitored System

The monitored system is considered to consist of the balance, the standard operating procedure, the laboratory environment, the check standard or control standard, the operator, and any other sources that contribute to the variance or bias of the measurement data. Any of the above that can be considered to be constant or negligible contributors to the variance may be consolidated and monitored by a

single control chart. Any that cannot be so considered (for example: different standard, different balance, different SOP) typically require separate control charts.

The variability of balance precision that is load dependent must be considered. For many balances, precision is a function of load, and a distinct control chart is required (according to the SOP) for every load tested. An F-test may be used to assess the standard deviations at varying loads to minimize the number of charts that are required in the laboratory when multiple loads have no significant difference in the standard deviation of the measurement process. In the case of SOP 5 or 28 (NISTIR 5672) check standards are incorporated into the measurement process and designated charts will be required. Hence, control charts used for measurement assurance and evaluation of measurement uncertainty are generally satisfactory if developed using data from check standards at two or three intervals for each balance appropriately spaced within the range of balance use, or at least with one check standard for each decade. On balances where few nominal values (loads) are tested, a control chart should be established for each load.

In the event that a check standard is not used to monitor each reference or working standard that is used, more frequent monitoring or calibration intervals may be needed to assure the quality of laboratory measurement results.

3.2. Selection, Care, and Calibration of Check Standards

3.2.1. A check standard must be stable and is normally comparable to the reference standard or to the typical item submitted for calibration, depending on what is being monitored (standards or process). For lower order calibrations, the check standard should simulate the laboratory's reference or working standards to the extent feasible. It should be calibrated with an expanded uncertainty equal to or better than the precision of the process being monitored. All check standards should be cared for in the same way as reference standards to prevent their damage or deterioration. Lower order check standards should be recalibrated at regular intervals according to NISTIR 6969, Good Measurement Practice (GMP) 11 on Setting and Adjusting Calibration Intervals.

3.2.2. Select check standards for mass calibration procedures as shown in Table 1. Take care to consider balance load and variability as noted in section 3.1 to ensure that standard deviations of the measurement processes are consistent at different nominal values. Use the next larger standard deviation of the measurement process in uncertainty calculations for intermediate nominal values. When a check standard is not used at each nominal value, additional monitoring of the working standards is required.

Table 1. Recommended check standards for typical test situations.

Procedure	Range of Measurement	Check Standard(s)
Echelon III (Class F) SOP 7, 8	5000 lb to 0.001 lb or 2268 kg to 1 mg	2 to 3 values per balance OR 1 chart per load
Echelon II SOP 3, 4, 6, 7	5000 lb to 0.001 lb and 1000 kg to 1 mg	2 to 3 values per balance OR 1 check standard per decade
SOP 5	typically 1 kg to 1 mg	each nominal value incorporates a check standard
SOP 28	typically 1 kg to 1 mg	1 check standard per decade (e.g., 1 kg, 100 g, 10 g, 1 g, 100 mg, 10 mg, 1 mg)

3.3. Establishing Control Chart Parameters

3.3.1. The control chart parameters consist of the central line, the best estimate of the mean of measurements of the check standard, and control (or "action") and warning limits that represent probabilistic limits for the distribution of results around the central line. These parameters are evaluated on the basis of a reasonable number of initial measurements and updated as additional measurement data are accumulated.

3.3.2. To establish a new control chart, make at least seven (minimum number) and preferably 12 or more, independent measurements of the check standard under the same conditions that will be used to make routine measurements. No two measurements should be made on the same day and the time of day should be varied as would be typical during routine laboratory operations. This is necessary to estimate the long-term standard deviation to the extent feasible. To make statistically valid decisions or calculate uncertainties based on this data, 25 to 30 points are necessary.

Calculate the mean, $\bar{x}$ and the estimate of the standard deviation, s in the conventional manner.

Establish the control chart parameters as follows:

Central Line $= \bar{x}$
Upper Control/Action Limit $= \bar{x} + 3s$
Upper Warning Limit $= \bar{x} + 2s$
Lower Warning Limit $= \bar{x} - 2s$
Lower Control/Action Limit $= \bar{x} - 3s$

Control chart parameters for Echelon III (Class F or other) may be completed as follows to track practical limits:

Central Line	$= \bar{x}$
Upper Control/Action Limit	$= \bar{x}$ *+ 1/4 tolerance*
Upper Warning Limit	$= \bar{x}$ *+ 1/10 tolerance*
Lower Warning Limit	$= \bar{x}$ *- 1/10 tolerance*
Lower Control/Action Limit	$= \bar{x}$ *- 1/4 tolerance*

3.3.3. Updating Control Chart Parameters

Update control chart parameters when a significant amount of additional data is available or when the previously determined parameters are no longer pertinent due to changes in the system.

Note: Ordinarily, updating is merited when the amount of new data is equal to that already used to establish the parameters in use, or when at least seven additional data points have been recorded.

Calculate $\bar{x}$ and s for the new set of data and examine for significant differences from the former using the t-test and F-test, respectively. If the tests fail and results are significantly different, determine the reason for the difference, if possible, and decide whether corrective action is required. If data does not agree within statistical limits, establish new parameters using the most recent data and note the reasons for not using previous data or correct the causes of variation. If no significant differences between the data sets are found, pool all data and calculate new control chart parameters based on all existing data.

3.4. Frequency of Measurement

The check standard should be measured and plotted with sufficient frequency to minimize the risk of loss of data during the period from last-known-in to first-known-out of control condition. It is good practice to measure the check standard at least once during each period when a set of test measurements is made. For critical calibrations or those of highest accuracy, it is desirable to alternate measurements of test items and check standards, but for real-time evaluation it is preferable to incorporate the check standard in the calibration design as in SOP 5 or SOP 28.

Whenever there has been a long period of inactivity, it is good practice to make a series of measurements of the check standard and to plot the results on a control chart to demonstrate attainment of statistical control prior to resuming measurements with that specific calibration system.

Control charts should be updated as close to real time as feasible to effectively monitor the measurement process to prevent the possible release of questionable data that may result in the recall of laboratory work.

4. Use of Control Charts

4.1. Monitoring a Measurement Process

Use the following criteria to interpret control chart results.

4.1.1. If plotted points are stable and randomly distributed within the warning limits, decide that the system is in control. If the process is in control (and the process statistic is normal), 99.73 % of the points will fall within the control/action limits. Observations outside the limits, or systematic patterns within, should be investigated and corrected if appropriate or possible.

4.1.2. If a plotted point is outside the warning limits but within the control limits, investigate the presence of calculation errors. If none were made, re-measure the check standard. The re-measured value must be within the warning limits to merit the decision of "in control". If the results are not within limits, consider the measurement process "out of control". Reject all data obtained since last "in-control" measurement and take corrective action (hence “action” limit). Accept no further data until the system is demonstrated to be in-control as indicated by at least two successive measurements of the check standard within the warning limits.

If a plotted point is outside the control limits and arithmetically correct, the system is considered to be out of control. Data are rejected, corrective actions must be taken and re-attainment of statistical control demonstrated, as above, before data may be accepted.

4.1.3. Additional guidelines for the evaluation of control charts based on probability statistics should be used to evaluate the presence of drift, shifts, and possible bias. Examples for further evaluation include:

4.1.3.1. Any single point or series of points outside of three standard deviations (keeping in mind the probability that a three standard deviation limit could reasonably expect to allow three points out of one thousand to be outside these limits);

4.1.3.2. Two of the last three points above (or below) two standard deviations;

4.1.3.3. Four of the last five points above (or below) one standard deviation;

4.1.3.4. Eight consecutive points on one side of the mean or reference value;

4.1.3.5. Six points in a row trending up (or down); and

4.1.3.6. Fourteen points alternating up and down.

4.2 Monitoring a Reference Value

4.2.1. When a Reference Value for the check standard is outside of one standard deviation of the mean value, it may necessitate obtaining an updated calibration or evaluating the bias or deviation further to determine the cause and correct it. The deviation or offset must be considered with respect to the reported uncertainty for the reference value as well as the measurement process being used to evaluate the value. In some cases, such as with very large tolerances, a measurement process might be quite small compared to the offset, in which case the offset can be used as an uncorrected systematic error in the uncertainty calculations. Where tolerances are small or uncertainty requirements stringent, updated calibrations may be required.

Note: the reference value may exceed acceptable limits even when a measurement process appears statistically in control. When tolerances are large compared to process variability, reference value offsets may simply be noted.

4.3. Transfer of Measurement Statistics

4.3.1. Absence of a significant difference between the central line and the accepted value for the check standard may be considered as evidence of insignificant bias at the level of confidence of the statistical test used. This conclusion is valid, as long as the system remains in control. On occasion, small differences (less than one standard deviation) from unknown sources will be obvious over time and the value observed for the bias should be incorporated into the uncertainty per SOP 29.

4.3.2. The estimate of the standard deviation of the process, s_p, used to establish the control limits may be used to calculate confidence intervals for all pertinent measurements made while the system is in control. However, see SOP 29 for calculation of measurement uncertainty using the process variability, s_p. The value of the test weight is said to be within the limits

$$\overline{y} \pm U$$

where $\overline{y}$ represents the mean of the measurements on the test weight and $U = k * \sqrt{u_S^2 + s_P^2 + u_O^2}$, with the value of k determined by the confidence level required for the interval.

Appendix A - Checklist for Creation or Evaluation of Control Charts

This checklist may be used as design criteria or to assess the quality of control chart construction.

Control Charts cover entire scope (are available for each measurement parameter).	
Control charts have titles (or otherwise include)	
	Laboratory name or other identifying information
	SOP(s)
	Balance/Other equipment
	Standard ID, Check standard ID
	Nominal load/value
	Dates/chronology/time periods if not used on the x axis
	Legends if multiple series and extra information are plotted; to avoid confusion
Control charts have x and y axis with labels and	
	All measurement values have units of measure associated with them
Control charts have	
	Mean value (and units)
	Standard deviation (and units)
	Degrees of freedom or number of points noted (if not obvious or if small number)
	Alternative summaries of this information and suitable references (e.g., tables)
	Printed summary reports of control data with the charts
	Reference values and source and bias if appropriate/available
Control charts have limits that are based on	
	Statistical controls of
	Warning limits (i.e., two standard deviations) and
	Action/control limits (i.e., three standard deviations)
	Or specification limits (e.g., tolerances or smaller ratios of tolerances)
Good Items (on chart or in spreadsheet or database table summaries). Control charts have (when applicable and meaningful if not otherwise noted, e.g., in a table)	
	Tolerances: when applicable
	Uncertainties: for reference value, check standard, and the process output
	Equipment information: device readability, configuration (stability settings/timing)
	Standard information: calibration date and interval information
	Responsible staff: need on chart or in database
	Status of control: in control, out of control with latest date of review
	History: previous limits and history of the chart/data with F-test and/or t-test results

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SOP No. 29

Standard Operating Procedure for the Assignment of Uncertainty

1. Introduction

1.1. Purpose

Laboratories performing calibrations that meet ISO/IEC 17025 must report uncertainties in conformance with the *ISO Guide to the Expression of Uncertainty in Measurement (*hereafter called the *GUM)*. This SOP provides instructions for the laboratory to meet this requirement.

1.2. Prerequisites

1.2.1. Calibration certificates with valid uncertainties must be available for all standards.

1.2.2. Statistical data regarding the calibration measurement process must be available; preferably from measurement control programs within the laboratory.

1.2.3. Knowledge of the technical basis for the measurement is critical for completeness in uncertainty evaluation. This can be obtained through reference papers, reference procedures, brainstorming, experimentation, interlaboratory comparisons, cause and effect diagrams and the like. Each NIST SOP published in NISTIR 6969, 5672, and 7383 includes detailed uncertainty budget tables that may be used.

2. Methodology

2.1. Scope, Precision, Accuracy

Each measurement made in a laboratory has a corresponding uncertainty assigned to the calibration value. The uncertainty is directly related to the measurement parameter (scope), range of the measurement, the equipment or measurement process being used (affecting precision), and the standards available with associated uncertainties.

2.2. Summary

This uncertainty analysis process follows the following eight steps:

1) Specify the measurement process;
2) Identify and characterize uncertainty components;
3) Quantify uncertainty components in applicable measurement units;

4) Convert uncertainty components to standard uncertainties in units of the measurement result;
5) Calculate the combined uncertainty;
6) Expand the combined uncertainty using an appropriate coverage factor;
7) Evaluate the expanded uncertainty against appropriate tolerances, user requirements, and laboratory capabilities; and
8) Report correctly rounded uncertainties with associated measurement results.

Special methods for handling bias/errors and uncertainties associated with the use of multiple standards are addressed as well.

3. The Process of Measurement Uncertainty Estimation

3.1. Step 1. Specify the process.

Clearly specify the measurement process in question, including the item being measured and the input quantities upon which it depends. This will usually require a quantitative expression related to the process (i.e., a measurement equation). Where possible, you may reference an SOP or other method description along with the specific standards and measurement assurance process that is used to adequately complete this step.

3.2. Step 2. Identify and characterize uncertainty sources.

Identify all possible sources of uncertainty in a comprehensive list, characterizing them based on the evaluation method that will be used to quantify them (Type A, statistical methods or Type B, scientific judgment) and to categorize them based on their relatedness with something such as an uncertainty budget table.

Table 1. Example uncertainty budget table.

Uncertainty Component Description	Symbol	Source	Type (A or B)	Typical Distribution
Standard uncertainty from the measurement *process*	s_p	Process	A	Normal
Standard uncertainty for the *standards*	u_s	Cal Cert	B	See Cert
Standard uncertainty due to *other* factors	u_o	Analysis or References	B	Varies

Using the measurement equation that was identified in 3.1 provides a good starting point as do the detailed uncertainty budget tables provided in many procedures. All of the parameters in this expression may have an uncertainty associated with them. When there are discrete steps in the measurement process, additional uncertainties may be associated with each.

What follows are the most common uncertainties associated with metrological measurements. Keep in mind that this list is not exhaustive. Each item listed below is identified as a standard uncertainty, *u*, when determined using Type B methods of evaluation (technical judgment, theoretical assessments, reference items) and a standard uncertainty, *s*, when determined with Type A methods of evaluation (statistical methods). Each standard uncertainty is represented by a lower case variable and further defined by a subscript that is arbitrarily assigned and usually related to the source for ease in remembering that source.

3.2.1. Standard uncertainty from the measurement *process*, s_p, (Type A evaluation).

3.2.1.1. Standard deviation from a measurement assurance chart or control chart.

The value for s_p is obtained from the control chart data and the current knowledge that the measurements are in a state of statistical control. This must be ascertained by measuring at least one check standard during the course of the current measurements combined with data accumulated using the same process in previous measurements. The control chart data must reflect the measurement process being performed.

3.2.1.2. Standard deviation from a series of replicate measurements.

Measure a stable test object at least seven times, no two measurements of which should be made on the same day. Calculate the standard deviation in the conventional manner to obtain the standard deviation of the process, s_p, keeping in mind that it does not fully represent the measurement process under all typically encountered conditions and that additional uncertainty values may need to be addressed more fully.

Note: Repetitive measurements made on the same day estimate the short-term standard deviation of the process and may underestimate actual measurement variability.

3.2.1.3. Standard deviation when a process standard deviation is “zero”.

When the standard deviation of a measurement process is less than the resolution of the measuring instrument, *d*, the standard deviation must be assessed. Generally, the smallest standard deviation to be used in this instance may be estimated as 0.577 times the instrument resolution from the equation noted below, with *d* being the instrument resolution. Assess the standard deviation and use the larger of the calculated standard deviation or the value from this equation. (Some procedures will provide for this value to be reduced further through repetitive measurements (e.g., SOP 4 and SOP 5); however, analysis of laboratory measurement assurance check standard data must be performed and be acceptable, to reduce the standard deviation of the measurement process further.)

$$s_p = \frac{d}{\sqrt{3}}$$

3.2.2. Standard uncertainty for the *standards*, u_s (Type B evaluation).

3.2.2.1. When using standards calibrated by another laboratory.

The information for the standards comes from the calibration report, generally reported as an expanded uncertainty with its coverage factor (k). The expanded uncertainty is simply divided by the stated k value to obtain the combined uncertainty for the standard, u_c, which represents the u_s when used in your laboratory. When k is not equal to two, additional factors associated with the degrees of freedom must be included. See Appendix A to determine the degrees of freedom associated with coverage factors. Keep track of the degrees of freedom for later use.

3.2.2.2. When using a standard calibrated in your laboratory (Type B evaluation).

If the standard was calibrated in your own laboratory, calculate the combined standard uncertainty, u_c, at $k = 1$ and use that as the standard uncertainty for the standard, u_s.

3.2.2.3. When using more than one standard (Type B evaluation).

When more than one standard is used in a calibration, the standard uncertainty for each, u_{s1}, u_{s2}, u_{s3}, etc., is included in the RSS equation if the standards have had independent calibrations. Standards with independent calibrations are combined by standard root sum square methods.

When calibrations are performed at the same time with the same reference standards, or when a standard is used multiple times, the standards are likely considered dependent, so the standard uncertainties are added ($u_{s1} + u_{s2}$) to determine a value to represent u_s. (This is the case with two 1 kg standards that were calibrated at the same time using a weighing design and subsequently used together as standards (restraints) in a weighing design. It is also the case when a 25 gal standard is used four times to calibrate a 100 gal standard.) There are also circumstances where both approaches are used to combine uncertainties from multiple combinations of standards.

3.2.3. Standard uncertainty due to *other* factors, u_o (Type B evaluation.)

These are factors related to the measurement equation, but distinct from the standard uncertainties associated with the process and the standards. These items are often much smaller in a well-controlled process than the standard uncertainties associated with the process and the standards. Examples are given in the uncertainty budget tables in each calibration SOP. Each component that is considered is included as an additional standard uncertainty u_{o1}, u_{o2}, u_{o3}, etc., and included in the RSS equation when data shows these factors to be significant. Documentation of the assessment of each component must be maintained to complete the documentation required by the specific calibration procedure and ISO/IEC 17025.

Additionally, the laboratory should include any other components that are considered significant. An estimated standard uncertainty that impacts the least significant digit in the combined uncertainty is considered significant. Additional uncertainties are addressed in the uncertainty budget tables provided in the SOPs.

3.2.4. Standard uncertainty due to factors *unrelated* to the measurement process per se, u_u.

These are factors that may be related to characteristics of the items being tested or of the standard and are usually minimized in well-known and controlled measurement processes. Review the uncertainty budget tables in each SOP or applicable international/national procedures or reference papers for more information.

3.2.5. Special uncertainties from other sources (Type B evaluations). Includes bias or unidentified errors.

It is a general requirement of the GUM that corrections be applied for all recognized and significant systematic effects and potential errors. Where a correction is applied based on a bias, an estimate of the associated uncertainty must then be included in the uncertainty analysis. Due to the various approaches present in the metrology system, several examples and possible approaches are presented in the section on calculating the combined or expanded uncertainties. At this stage, a determination must be made with regard to 1) identifying cause and 2) level of significance.

3.2.5.1. Identifying bias (offsets) and cause.

Bias or measurement offsets from reference values are often noted on control charts when an independent reference value is available and from results in proficiency testing. Control chart values are obtained over time and usually have more degrees of freedom than results from one or a few proficiency testing results. Therefore, noted offsets on control charts are

more reliable estimates of bias when independent reference values are available. Evaluation of data from multiple sources should be considered whenever available (e.g., a recent calibration, a proficiency testing result, and control chart data assessed as a part of an integrated system).

If the cause of bias or error can be identified, it is usually corrected or applied to the measurement equation. In some cases, it is not possible to unarguably define the cause without exhaustive studies that provide little benefit. In those cases, the significance level *must* be evaluated before incorporating this type of an uncorrected systematic error in the calibration uncertainties.

3.2.5.2. Significance level.

When there is little to be gained from exhaustive studies on the measurement process to identify bias or potential errors, a test of significance may be conducted to determine alternative approaches for incorporating the bias into the uncertainty calculations.

In deciding whether a known bias can reasonably be included in the uncertainty, the following approach may be followed:

3.2.5.2.1. Estimate the combined uncertainty without considering the relevant bias.

3.2.5.2.2. Evaluate whether or not the bias is less than the combined uncertainty (i.e., $bias < \sqrt{u_s^2 + s_p^2 + u_o^2}$).

3.2.5.2.3. If the bias is less than the specified limit, it may be included in the uncertainty using one of several approaches that must clearly be communicated in the report.

3.2.5.2.4. If the bias is larger than the specified limit, the error must be investigated further and corrected prior to providing calibration data.

If the deviations show that a standard is out of control, it should not be used for calibration until corrective action has been taken and the value for the standard is verified as being within criteria limits.

$$criteria\ limit: \left| \bar{x}_{lab} - x_{ref} \right| < u_c$$

If these differences are smaller than the criteria limits, investigation and corrective action may be unrealistic. If the deviations are less than the surveillance limits shown above,

and corrective action is not taken, the deviations may be included in the uncertainty statement following one of several options given in the following section provided that the resulting uncertainty meets the needs of the customer or application. In all cases, the method used to incorporate bias must be clearly reported.

3.2.5.3. Option 1. Adding the bias to the expanded uncertainty (e.g., used in PMAP software). In this case, the bias is simply added to the expanded uncertainty and is reported as such.

$$U + \text{bias} = (u_c * k) + \text{bias}$$

3.2.5.4. Option 2. When uncertainties for the laboratory data and the reference data are considered equivalent (e.g., laboratory data is compared to data from another laboratory having equivalent precision) the equation below may be used.

In this case, a rectangular distribution is considered where the value might possibly be anywhere within the range shown for each laboratory data point. This method is referenced in section 4.6 of NIST Technical Note 1297. This approach may also be used in the case where a standard is predictably drifting with use over time. In this case, a mid-range value is chosen and u_d (uncertainty for *differences*) is calculated as follows:

$$u_d = \frac{\text{bias}}{2}\frac{1}{\sqrt{3}} \quad \text{or more simply: } 0.29\,\text{d, where d is the bias}$$

3.2.5.5. Option 3. When uncertainties for the laboratory data are considered secondary to a reference value (e.g., the difference between the laboratory data and data from a higher level calibration with smaller uncertainties) the equation below may be used.

In this case, a reference value is given precedence over the laboratory data and a mid-range value is not chosen. The extreme value is more probable. In this case, the bias is treated as an uncorrected systematic error and the following equation may be used:

$$u_d = \frac{\text{bias}}{\sqrt{3}} \quad \text{or more simply : } 0.577\ \text{d, where d is the bias}$$

3.3. Step 3. Quantify uncertainty estimates

All uncertainty estimates identified in the previous step must be quantified in units that represent the measured values. Type A methods of evaluation usually provide quantified estimates in the units of interest.

Type B methods of evaluation may be conducted with spreadsheets using the basic expression identified in the SOP or identified when the process was specified. Scenario testing can be done to determine the impact and quantify specific variables on the final measured quantity. The knowledge gained in this step often proves useful in identifying potential areas of improvement especially if contributing factors are graphed in a histogram or Pareto chart.

3.4. Step 4. Convert all factors to standard uncertainties

In cases where the uncertainty factors were determined statistically (Type A methods), the standard deviation is used to represent the standard uncertainty. In other cases, estimates must be made to ensure that the quantified uncertainties represent "one-standard-deviation" values or a $k = 1$ coverage level.

The appropriate distribution factor must be used when converting estimated uncertainty values to standard uncertainties. According to NIST Technical Note 1297, a rectangular distribution is generally used when detailed information about the distribution is unknown.

$$u_n = \frac{\text{value}}{\sqrt{3}}$$

3.5. Step 5. Calculate the combined uncertainty

The combined standard uncertainty, u_c, includes the standard uncertainty reported for the standards used, u_s, the standard uncertainty of the measurement process, s_p, the standard uncertainty from other sources, u_o, which includes all other factors the laboratory considers significant, the standard uncertainty due to factors related to the measured item but unrelated to the measurement process, u_u, and finally, the standard uncertainty due to bias or differences, u_d, when u_d is included. The standard uncertainties are usually combined using the root-sum-of-the-squares (RSS) method as follows:

$$u_c = \sqrt{s_p^2 + u_s^2 + u_o^2 + u_u^2 + u_d^{\,2}}$$

Table 1. Symbol descriptions.

Symbol	Description
U	Expanded uncertainty (this is represented with an upper-case U)
u_c	combined standard uncertainty
s_p	standard uncertainty (standard deviation) of the "process"
u_s	standard uncertainty of the "standard"
u_o	standard uncertainty of "other factors"

Symbol	Description
u_u	standard uncertainty of factors “unrelated” to the measurement process
u_d	standard uncertainty of “differences” (may be treated in different ways)
k	coverage factor

3.6. Step 6. Calculate the expanded uncertainty

The combined standard uncertainty is then multiplied by a coverage factor, *k*, based on the degrees of freedom, to provide a level of confidence of approximately 95 % or 99 %, respectively (depending on what is required by the customer, but most often 95 %). The equation used to determine the expanded uncertainty is as follows:

$$U = u_c * k$$

where $k = 2$ a value from the table in Appendix A, taken from the column 95.45. When there are a small number of degrees of freedom, the coverage factor must be determined from a statistical table such as provided in the Guide to the Expression of Uncertainty in Measurement or NIST Technical Note 1297 (See Appendix A).

Situations where the degrees of freedom associated with the value for a standard in the traceability chain require special care to ensure that an appropriate coverage factor is used for calculating the uncertainty of subsequent reported values. When there are a small number of degrees of freedom used to determine the standard deviation of the process and/or the *k* value from the calibration report represents a small number of degrees of freedom, the *effective degrees of freedom* must be used to determine an appropriate coverage factor. Use the equation provided in NISTIR 6969, Section 8 or the Welch-Satterthwaite equation provided in NIST Technical Note 1297 or the Guide to the Expression of Uncertainty in Measurement.

3.7. Step 7. Evaluate the expanded uncertainty

The expanded uncertainty must be evaluated. Evaluation may be against established criteria such as tolerance limits, customer requirements, and/or calibration and measurement capabilities listed on the laboratory scope. For example, the specifications for mass standards clearly state that the expanded uncertainty must be less than 1/3 of the tolerance.

3.8. Step 8. Report the uncertainty

Once the uncertainty has been calculated, round the value according to GLP 9, report the value, and include a statement such as the following is reported:

The combined standard uncertainty includes the standard uncertainty reported for the standard, for the measurement process, and for any observed deviations from reference values (e.g., from NIST), which are less than surveillance limits. The combined standard uncertainty is multiplied by *k*, a coverage factor of (insert value used) to give the expanded uncertainty which defines an interval with an approximate 95 % level of confidence.

The value selected for the coverage factor must be appropriate for the degrees of freedom available and the desired level of confidence associated with the uncertainty as noted in Step 7.

Appendix A

Value of $tp(\nu)$ from the t-distribution for degrees of freedom ν that defines an interval $tp(\nu)$ to $+tp(\nu)$ that encompasses the fraction p of the distribution. Note: Table is taken from NIST Technical Note 1297. The column marked 95.45(a) is generally used to give the "approximate 95 % confidence interval."

Degrees of freedom ν	Fraction p in percent					
	68.27(a)	90	95	95.45(a)	99	99.73(a)
1	1.84	6.31	12.71	13.97	63.66	235.80
2	1.32	2.92	4.30	4.53	9.92	19.21
3	1.20	2.35	3.18	3.31	5.84	9.22
4	1.14	2.13	2.78	2.87	4.60	6.62
5	1.11	2.02	2.57	2.65	4.03	5.51
6	1.09	1.94	2.45	2.52	3.71	4.90
7	1.08	1.89	2.36	2.43	3.50	4.53
8	1.07	1.86	2.31	2.37	3.36	4.28
9	1.06	1.83	2.26	2.32	3.25	4.09
10	1.05	1.81	2.23	2.28	3.17	3.96
11	1.05	1.80	2.20	2.25	3.11	3.85
12	1.04	1.78	2.18	2.23	3.05	3.76
13	1.04	1.77	2.16	2.21	3.01	3.69
14	1.04	1.76	2.14	2.20	2.98	3.64
15	1.03	1.75	2.13	2.18	2.95	3.59
16	1.03	1.75	2.12	2.17	2.92	3.54
17	1.03	1.74	2.11	2.16	2.90	3.51
18	1.03	1.73	2.10	2.15	2.88	3.48
19	1.03	1.73	2.09	2.14	2.86	3.45
20	1.03	1.72	2.09	2.13	2.85	3.42
25	1.02	1.71	2.06	2.11	2.79	3.33
30	1.02	1.70	2.04	2.09	2.75	3.27
35	1.01	1.70	2.03	2.07	2.72	3.23
40	1.01	1.68	2.02	2.06	2.70	3.20
45	1.01	1.68	2.01	2.06	2.69	3.18
50	1.01	1.68	2.01	2.05	2.68	3.16
100	1.005	1.660	1.984	2.025	2.626	3.077
∞	1.000	1.645	1.960	2.000	2.576	3.000

(a)For a quantity z described by a normal distribution with expectation μz and standard deviation σ, the interval $\mu z \pm k\,\sigma$ encompasses p = 68.27, 95.45, and 99.73 percent of the distribution for k = 1, 2, and 3, respectively.

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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).

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SOP No. 33

Standard Operating Procedure for Calibration of Weight Carts

1. Introduction

1.1. This Standard Operation Procedure (SOP) describes the procedure to be followed for the calibration of weight carts used to test livestock and vehicle scales.

1.2. Prerequisites

1.2.1. Facility. Verify that the laboratory facilities meet the following minimum conditions to meet the expected uncertainty possible with this procedure.

Table 1. Environmental conditions.

Echelon	Temperature Requirements During a Calibration	Relative Humidity (%)
III	Lower and upper Limit: 18 °C to 27 °C Maximum changes: ± 5 °C / 12 h and ± 3 °C / h	40 to 60 ± 20 / 4 h

1.2.2. Balance/scale. Verify that the balance, scale, or load cell is in good operating condition with sufficiently small process standard deviation as verified by a valid control chart or preliminary experiments to ascertain its performance quality. The expanded uncertainty (k = 2) must be less than one-third of the applicable tolerance and balance operating characteristics must be evaluated against this requirement prior to calibration.

1.2.2.1. If a scale or load cell is used for the calibration that is not a permanent piece of equipment in the calibration laboratory, appropriate verification and repeatability statistics must be obtained prior to a calibration to determine suitability and acceptability for calibration. Records must be maintained of this verification. Minimum verification includes an increasing and decreasing load test to at least the capacity of the weight cart, a shift test, a sensitivity test at the test load, and evaluation of the repeatability based on a minimum of 7 repeated weighings (following the same SOP to be used to test the weight cart).

1.2.3. Standards. Ensure the availability of suitable working standards or recently calibrated (and unused) field standards in quantities up to the capacity of the weight cart(s) that will be calibrated. All standards must have known conventional mass values with valid metrological traceability to the SI, which may be through laboratory working standards to the National Institute of Standards and Technology.

Standards must be evaluated to ensure that standard uncertainties for the intended level of calibration are sufficiently small. Reference standards should not be used to routinely calibrate customer standards using this procedure. The expanded uncertainty (k = 2) must be less than one-third of the applicable tolerance and evaluated prior to calibration. The contribution of the mass standards must be assessed prior to calibration and Class F mass standards without corrections or with uncertainties greater than 1/3 of the tolerance will not be acceptable.

1.2.4. Staff. Verify that the operator is experienced in precision weighing techniques and has had specific training in SOP 8, SOP 4, SOP 29, and GMP 10. Safety precaution: weight carts should be calibrated with at least two people present.

2. Methodology

2.1. Scope, Precision, Accuracy

This method is applicable to lower echelon mass calibration (tolerance testing) of weight carts, provided that the uncertainty requirements can be met. The achievable precision using this procedure is appropriate, provided the expanded uncertainty of the measurement is no more than one-third of the permissible tolerance of the weight cart. The accuracy achievable with this procedure depends on the accuracy of the calibration of the working standards and the precision of the comparison equipment/devices chosen and the procedure selected (e.g., SOP 8 or SOP 4).

2.2. Summary

A weight cart is calibrated by substitution with standards having known values, using weighing equipment that has been evaluated as having sufficient resolution and repeatability, to maintain uncertainty to tolerance ratios needed in legal metrology. Modified or double substitution methods are used (SOP 8, SOP 4) and are modified to obtain the replicate observations needed for measurement assurance and determination of the calibration uncertainty.

2.3. Equipment /Apparatus/Assistance

2.3.1. A balance, scale, or load cell with suitable resolution and repeatability, in good repair.

2.3.1.1. General guidelines regarding selection of weighing equipment must include consideration of the device repeatability to determine the achievable standard deviation of the measurement process. To ensure that appropriate uncertainty to tolerance ratios are maintained, the following guidance is provided:

2.3.1.1.1. When using Modified SOP 8:
- For a 1000 lb to 2000 lb cart, the maximum division size should be 0.01 lb or less.
- For a 5000 lb cart, the maximum division size should be 0.05 lb or less.

2.3.1.1.2. When using Modified SOP 4:
- For a 1000 lb to 2000 lb cart, the maximum division size should be 0.05 lb or less.
- For a 5000 lb cart, the maximum division size should be 0.1 lb or less.

2.3.1.2. Some balances will require suitable supports to be mounted on the scale deck (platform) to receive the weight cart dimensions without damage to the scale or weight cart. Supports must be appropriately stable and sturdy.

2.3.2. Standards and correction weights, meeting NIST Class F tolerances with current Calibration Reports and known values and uncertainties (as listed with prerequisites).

2.3.3. Hand tools such as the following are needed: wrenches; screw driver; wire cutters; lead and wire seals, and seal press.

2.3.4. Means to move the weight cart safely. Weight carts are large, bulky items and are awkward to move safely. Appropriate safety precautions must be followed in the laboratory. Movement of liquid powered fuel carts is usually not done using the power system of the carts because exhaust from gasoline or diesel powered carts is considered unsafe in the laboratory; if no other options exist, suitable air handling must be available. Use of forklifts to move the carts should only be considered when other options are unavailable and extreme care must be taken. Use of a hoist may provide adequate means to move the cart, however, adequate care must be taken to prevent tipping the cart or bumping into other items in the laboratory as the cart is lifted and moved.

2.3.5. A basic knowledge of weight carts, including the braking system and operating controls is required. Only qualified personnel should operate and inspect the weight cart. Verification that the brakes are operative prior to movement of the cart is essential.

2.3.6. Suitable adjustment material must be available for placement in the adjusting compartments; lead or steel shot is recommended.

2.3.7. Inspection checklist (See Appendix A). Weight carts are not considered Class F standards due to the extent of motorized and mechanical parts and fluids; therefore, special care must be taken in their calibration and use. An inspection checklist must be completed for each new cart prior to placing it into service. The checklist becomes a permanent part of the records for each weight cart and must be available for review prior to each use or calibration.

2.3.8. For safety reasons, two people should be present to conduct a calibration of a weight cart.

3. Fundamental Considerations

3.1. Weight carts must be properly cleaned and painted, with all repairs and maintenance completed (as needed) prior to calibration. Obtaining actual "as found" values for reverse traceability is not possible with this procedure. Obtaining "as found" values may be achieved by performing a calibration before and after repairs and maintenance are completed. Alternatively, see the "Recommended Method for Intermediate Verification of Mass Values for Weight Cart Users" included as Appendix B.

3.2. Weight carts must be completely dry, with no ice or moisture on the surface or under carriage of the cart.

Experience has shown that a light coating of frost can cause errors in excess of the tolerance for the cart. In heated facilities, the cart must be allowed sufficient time to warm to prevent condensation.

Table 2. Stabilization Time.

Temperature Range 22 ºC (lab temp)	Temperature Range 72 ºF (lab temp)	Minimum Equilibration Time*
± 5 ºC, 17 ºC to 28 ºC	± 10 ºF, 62 ºF to 82 ºF	4 hours
< 17 ºC and > 28 ºC	< 62 ºF and > 82 ºF	24 hours/overnight
* deviation is allowed if the known cart temperature is at the lab temperature ± 2.5 ºC (5 ºF).		

3.3. Weight carts that repeatedly demonstrate excessive "as found" out-of-tolerance conditions from the previous calibration date, may be required to be submitted on a more frequent basis; with every consideration being given to determine the cause of the variance.

4. Pretest Weight Cart Inspection

4.1. Complete the Weight Cart Inspection Checklist that is included in Appendix A. The Inspection Checklist is an integral part of the Calibration Report of a weight cart and must be transported with each weight cart and verified prior to each use or calibration. Note and ensure completion of any needed repairs or maintenance prior to calibration.

5. Procedure

5.1. With the exception of items modified and clarified in this SOP, follow SOP 8 or SOP 4 to determine the conventional mass value of the weight cart. Follow good laboratory practices such as properly exercising the balance prior to calibration.

5.2. SOP 8 Modifications.

5.2.1. Repeat the intermediate weighing of the weight cart as if multiple weights were being verified. Obtain at least 3 observations that are taken with a release/removal of the weight from the sensing device. Determine the calculated differences per SOP 8 and calculate the range between the largest and smallest numerical mass value differences. Record the range values in a suitable control chart for weight cart calibrations.

5.2.2. Mass adjustments to the cart may be made at the end of exercising the balance or after the first observation. The cart must be adjusted as close as possible to zero error. The first reading then becomes a "zero" difference when compared to the standards (the correction values of the standard must be considered). Adjustments must be made quickly to avoid errors due to balance drift or shifting. In the event that balance drift or shifts are noticed, the 3 replicate observations should be made after the adjustment is completed.

5.2.3. Care must be taken to ensure that the center of the mass of the weight cart is centered on the balance pan. By rotating the cart by 180 degrees and remeasuring it, off-center loading factors may be considered as a part of standard deviation.

5.3. SOP 4 Modifications.

5.3.1. Repeat the double substitution procedure 3 times. Determine the calculated differences per SOP 4 and calculate the range between the largest and smallest differences. Record the range information in a suitable control chart for weight cart calibration range values.

5.3.2. Mass adjustments to the cart may be made at the end of exercising the balance or after the first double substitution. The cart must be adjusted as close as possible to zero error. The first reading then becomes a "zero" difference when compared to the standards (the correction values of the standard must be considered). Adjustments must be made quickly to avoid errors due to balance drift or shifting. In the event that balance drift or shifts are noticed, the 3 replicate double substitutions should be made after the adjustment is completed.

5.3.3. Care must be taken to ensure that the center of the mass of the weight cart is centered on the balance pan. By rotating the cart by 180 degrees and remeasuring it, off-center loading factors may be considered as a part of standard deviation.

6. Calculations

6.1. Follow calculations as described in SOP 4 or SOP 8 as appropriate.

7. Tolerance

7.1. Tolerances are established in NIST Handbook 105-8. Weight carts must be adjusted as close as possible to zero error during calibration. The expanded (k=2) combined uncertainty of the calibration must be less than one-third of the tolerance specified in Handbook 105-8.

7.2. The values of weight carts and their tolerance status are issued and evaluated on the basis of Conventional Mass (previously called Apparent Mass versus a reference density of 8.0 g/cm^3 at 20 °C). If a reference density other than 8.0 g/cm^3 is used, the reference density must be reported. No correction for the effect of air buoyancy is general made unless stated on the test report.

7.3. Tolerances for weight carts (See also NIST Handbook 105-8).

Nominal Mass (lb)	**Tolerance (± lb)**
1000	0.25
2 000, 2 500	0.50
3 000, 3 500	1.00
4 000, 4 500	1.25
5 000, 5 500	1.50
6 000 +	2.00

8. Measurement Assurance

8.1. Adequate measurement assurance is a required component of every quality system and uncertainty analysis, but maintaining a surrogate weight cart for use in the laboratory as a check standard is impractical.

8.2. Three repeated observations are made and the standard deviation of the values or the range of values (largest mass difference – smallest mass difference) is entered into a control chart. The pooled standard deviation of the process is used or the pooled mean range value is used to estimate a standard deviation of the process. When plotting each new range value, verify that it is within previously accepted limits.

8.3. Alternatively, 7 to 12 repeated observations are made (following the same SOP to be used to test the weight cart) and a short-term standard deviation of the process is calculated each time a weight cart is calibrated. A minimum of 7 replicate weighings may also be used in the event that a balance is used that is not permanent equipment of the laboratory.

9. Assignment of Uncertainty

The limits of expanded uncertainty, U, include estimates of the standard uncertainty of the mass standards used, u_s, and estimates of the standard deviation of the measurement process, s_p. These estimates should be combined using the root-sum-squared method (RSS), and the expanded uncertainty, U, reported with a coverage factor of two (k=2), to give an approximate 95 percent level of confidence. See SOP 29 for the complete standard operating procedure for calculating the uncertainty.

9.1. The expanded uncertainty for the standards, U, are obtained from the calibration report. The combined standard uncertainty, u_c, is used and not the expanded uncertainty, U, therefore the reported uncertainty for the standards will usually need to be divided by the coverage factor k.

9.1.1. A combination or summation of standards is typically needed for the calibration of weight carts. The dependence or independence of the calibration of such standards must be determined prior to combining the uncertainties associated with the standards. In the case of dependent standards (calibrated against the same working standard), the u_s values are summed by addition prior to including them in a root sum square calculation: $(u_{s1} + u_{s2} + \ldots u_{sn})$. In the case of independent standards, the u_s values are summed by root sum square: $(u_{s1}^2 + u_{s2}^2 + \ldots u_{sn}^2)^{0.5}$. All u_s values must represent one standard deviation prior to combination.

9.2. Standard deviation of the measurement process, s_p, is obtained by using the pooled standard deviation of similar size carts over time or by

calculating the estimated standard deviation of the process based on the mean range value calculated from applicable range charts.

9.3. Buoyancy corrections are not generally needed at the uncertainty level this procedure is designed for. An uncorrected systematic error should be included for the uncorrected magnitude of the air buoyancy correction, following NISTIR 6969, SOP 2; this value is treated as a rectangular distribution.

9.4. Additional components due to balance drift or sensitivity errors may need to be included per SOP 8.

9.5. The expanded uncertainty, U, must be $\leq 1/3$ of the tolerance applicable as per NIST Handbook 105-8.

10. Calibration Report

Prepare a Calibration Certificate according to SOP 1. A sample calibration certificate is included in Appendix A. The Inspection Checklist is an integral part of the calibration certificate.

Appendix A
Sample CalibrationCertificate
For
Weight Cart

Test Number: **Date Issued:** **Date of Calibration:**

Submitted by:

Manufacturer: **Date of Manufacture:**

Model Number: **ID/SN Number:**

Calibration Values:

As Found Conventional Mass* (lb)	As Left Conventional Mass (lb)	Uncertainty (lb)

** The Conventional Mass is the mass determined/calculated by weighing in air of reference density 1.2 mg/cm^3 against reference weights of density 8.0 g/cm^3. Buoyancy corrections are considered negligible and were not made unless otherwise stated.*

Conditions of Test & Traceability:

The above weight cart was compared with standards of the State of XXXX, which are traceable to the National Institute of Standards and Technology (NIST) and have known values. The assigned test number shown above provides documented evidence for measurement traceability.

Test method: {Enter SOP 4 or 8 as applicable} NISTIR 6969, Selected Procedures for Mass Calibrations. {Enter publication date}
Balance:
Temperature:
Pressure:
Relative Humidity:

Components of Uncertainty:

The uncertainty is reported as an expanded uncertainty at an approximate 95 % confidence interval ($k = 2$) and calculated according to SOP 29 which is compliant with the ISO/IEC Guide to the Expression of Uncertainty in Measurement. Components included in the calibration uncertainty are the uncertainty associated with the standards and the standard deviation of the measurement process as determined through repeated observations or other suitable range chart for this measurement process. Factors included on the inspection checklist have not been included in the calibration uncertainty. However, factors on the checklist may contribute measurement errors that are significant if not properly maintained during use.

Inspection Checklist:

The weight cart was cleaned and painted and allowed to come to thermal and environmental equilibrium in the laboratory prior to calibration. The weight cart was adjusted (as needed and noted above) as close as possible to zero error. All fluid levels were adjusted as close as possible to the full/reference marks and sealed. Liquid levels must be maintained as close to reference levels as possible during use. The fluid levels include:
_____Engine Oil;
_____Hydraulic Fluid (sealed);
_____Battery Water Level (sealed battery); and
_____Liquid Fuel {Enter gasoline or diesel here} (note reference mark).
The attached Inspection Checklist is an integral component of this Calibration Report and a copy must be maintained with the cart and reviewed prior to use.

Any maintenance, repairs, replacement of parts, or damage to weight cart or its components will likely result in an out-of -tolerance condition; therefore, maintenance or replacement of components such as batteries, tires, filters, or other items listed on the checklist, require calibration of the weight cart prior to subsequent use.

Tolerance:

Reference: 2001 Draft NIST Handbook 105-8, Specifications and Tolerances for Weight Carts. The applicable tolerances are as follows:

Nominal Mass (lb)	Tolerance ± (lb)
1000	0.25
2 000, 2 500	0.50
3 000, 3 500	1.00
4 000, 4 500	1.25
5 000, 5 500	1.50
6 000 +	2.00

For carts intermediate between these values, the next smaller tolerance is applied.

_____
Authorized Signature

Inspection Checklist for Weight Cart

Test Number: Date of Inspection:

Manufacturer: Date of Manufacture:

Model Number: ID/SN Number:

_____Nominal Mass of Weight Cart: _____ Suitably marked: Yes/No
_____Powered by: Electric/Generator_____ Diesel_____ Gasoline _____
_____Fluid Levels: Engine Oil _____
Hydraulic Fluid _____ Sealed: Yes/No
Battery _____ Sealed: Yes/No
Liquid Fuel _____ Reference line present: Yes/No
_____Fluid drain tubes extend beyond the body of the cart: Yes/No
_____Number of axles: _____
_____Number/Size of Tires: _____
_____Sealed wheel bearings: Yes/No
_____Drain holes present in locations where water may accumulate: Yes/No
_____Weight restraint railing permanently fixed and solid: Yes/No
_____Adjusting cavity accessible: Yes/No
_____Adjusting cavity approximate capacity: _____lb
_____Adjusting cavity sealed: Yes/No
_____Service brakes functioning properly: Yes/No
_____Parking brakes functioning properly: Yes/No
_____Remote control functioning properly: Yes/No

_____General condition at time of calibration (note any accumulated dirt/debris, damage, loose parts, or evidence of tampering or unauthorized entry of seals).

_____

_____

_____List and report any repair and maintenance performed, parts replaced, etc., Leaks repaired, new battery, carburetor, exhaust system, wheels changed, welding performed, etc.. Include any comments or changes since the last calibration.

_____

_____

_____

Appendix B

Recommended Method for Intermediate Verification of Mass Values for Weight Cart Users

1. Introduction

1.1. This Recommended Method is intended to provide an intermediate verification of weight cart mass values to obtain a rough estimate of an "as found" value to provide *adequate* evidence for or against the need for corrective action on scales that have been tested.

1.2. This method may be performed as often as needed during a calibration cycle as a preventive action due to the inherent instability of weight cart mass values. It should also be performed prior to repairs or maintenance to obtain and record adequate evidence of mass values and calibration status prior to the change of mass values resulting from repair and maintenance.

1.3. Records of all such intermediate tests should be retained with the Inspection Checklist and should be submitted with the completed Inspection Checklist when the weight cart is submitted for calibration.

1.4. This method may NOT be used as a substitute for a proper laboratory calibration according to SOP 33 and does NOT provide a traceable mass value with an appropriate calibration uncertainty. Recall that the error on the standard must be less than one third of the applicable tolerance applied to a scale under test. This level of verification is difficult and may be impossible to determine under field conditions. Only errors in excess of Handbook 44 requirements may be determined using this method.

2. Prerequisites

2.1. Facility. Intermediate verification tests are normally conducted under field or under maintenance shop/garage conditions rather than in a controlled laboratory environment. As such, the avoidance of extreme conditions of temperature, wind, and precipitation are critical. A rough estimate of conditions at the time of verification must be recorded when data is obtained.

2.2. Equipment. A suitable scale must be selected. This typically needs to be of better resolution than the scales being tested with the weight cart or as a minimum, a scale with the smallest resolution on which the cart may be used. A repeatability test, with a minimum of 3 repetitions is needed to

ensure that the mass values will repeat to within one-half of a scale division at the load of the weight cart.

2.3. Standards. Class F field standards are used as long as they are within the prescribed calibration cycle. A small Class F weight kit is used as correction weights to determine small errors and partial division values.

2.4. Staff. Staff must be trained in substitution calibration methods and be able to perform calculations associated with determining mass values.

2.5. The weight cart fuel levels, where liquid fuels are used, must be set at the reference mark on the fuel sight gauge.

3. Fundamental Considerations

Weight carts must be completely clean and dry, with no mud, ice or moisture on the surface or under carriage of the cart. Tires must be cleaned as well. (Values obtained under actual operating conditions may provide some insight to the user regarding the impact of maintenance and cleanliness of the weight cart).

4. Pretest Weight Cart Inspection

Complete the Weight Cart Inspection Checklist that is included in Appendix A.

5. Procedure

5.1. Establish scale repeatability with a minimum of three repetitions such that the scale repeats to within one-half of a division at the weight cart capacity.

5.2. Load Class F field standards onto the scale in the same location(s) where the weight cart will be placed to the same load as the stated conventional mass of the weight cart. Assign a mass value to this summation of standards based on the nominal values of the weights. The added mass values of these weights is the value for your standards. With the weights loaded on the scale deck, use small error weights to determine the break point of the nearest larger scale division.

5.3. Remove the large Class F field standards and leave the small error weights on the scale.

5.4. Load the weight cart onto the scale in the same location(s) where the weights were loaded. Add or remove the small error weights until the scale responds in the same way as the previously loaded mass standards to determine the break point of the same scale division.

6. Calculations

Calculate the difference between the error weights used with the Class F field standards and the error weights used with the weight cart. Because this is not an accepted calibration method, the observed difference in these values only represents an estimated mass value. (Error weights used with Class F standards – Error weights used with the weight cart = Observed Estimated Error).

7. Tolerance & Evaluation

The observed estimated error must be less than one third of the smallest tolerance applied to a scale on which the weight cart might be used. Corrective action is required if the observed mass of the weight cart exceeds the calibrated mass by more than one-third of the applicable tolerance.

8. Measurement Assurance

Measurement assurance is obtained through three replicate measurements.

9. Assignment of Uncertainty

An uncertainty is not calculated, estimated, or assigned with this intermediate verification method. It is not considered a valid calibration with evidence of traceability.

10. Calibration Report

A calibration report is not prepared for an intermediate verification. Recorded and observed data should be maintained.

Example of Intermediate Verification Procedure and Calculations

A. A 2000 lb weight cart is to be tested on a scale with 10 lb divisions.

B. When 2) 1000 lb Class F field standards are placed on the scale, the scale reading repeats to within one-half of a division when 3 replicate weighings are performed (weights must be moved on and off the scale or the scale arrested and released to determine this repeatability). The scale reads 2000 lb for each test.

C. Error weights are slowly placed on the scale deck in increments of 0.1 lb and the addition of 5.5 lb causes the scale to read the next larger division of 2010 lb.

D. The 2) 1000 lb Class F field standards are removed and the 2000 lb weight cart is placed on the scale deck, leaving the previous 5.5 lb error weights. Error weights of 6.5 lb are needed for the scale to break to 2010 lb with the weight cart on the scale deck (an additional 1.0 lb was added).

E. Error weights used with Class F standards = 5.5 lb

F. Error weights used with the weight cart = 6.5 lb.

G. Error weights used with Class F standards – Error weights used with the weight cart = Observed Estimated Error; 5.5 lb – 6.5 lb = - 1 lb.

H. Evaluation: the smallest tolerance on the scale will be one-half of a division, or 5 lb. One third of 5 lb is 1.7 lb. The error on standards used on the scale must be less than this level. The observed error on the cart of – 1.0 lb is less than 1.7 lb and corrective action for previous scale tests is not needed at this time. The –1 lb value is recorded on the Inspection Checklist as an Intermediate Verification.

I. If the cart has been damaged or if repairs to the cart are needed, calibration is required at this time.

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SOP 34

Standard Operating Procedure[1] for the Selection and Use of Sensitivity Weights and Tare Weights in Weighing Procedures

1. Introduction

Mass calibration procedures are based on comparing the unknown mass, *X*, to a standard mass, *S*, utilizing the balance as a comparator. This comparison relies on the accuracy of balance indications. When balance indications are not accurate enough for precision mass calibrations, and they drift with time, appropriate procedural adjustments are required. Drift can often be assumed to be linear over a short period. Concerns over balance inaccuracy and drift result in two possible causes of errors in mass determination procedures. Inaccuracy of the balance indications can be corrected by incorporating a sensitivity weight in the procedure that calibrates the range of use of the optical scale (mechanical balances) or of the digital indications (electronic balances). Errors due to drift can be minimized by using the correct comparison method, selecting a suitable sensitivity weight, and by consistent timing within the procedure. The proper selection of procedures (GMP 12), the adherence to those procedures, and equal time intervals between weighing operations will allow the measured difference between *X* and *S* to be corrected for inaccuracy of the balance indications and for balance drift.

Mass comparison procedures rely on the unknown and standard masses to be nominally equal. When the mass standards are not near to each other, tare weights need to be used to bring them closer together. Tare weights in this case function as additional mass standards which will essentially be treated as additional mass standards in summation.

1.1. Purpose

The following practice will guide you through the process of selecting and using a correct sensitivity weight and/or tare weight(s) for mass determination procedures.

1.2. Prerequisites

1.2.1. Valid calibration certificates with appropriate values and sufficiently small uncertainties must be available for all of the sensitivity weights and tare weights used in a calibration. All mass standards must have demonstrated metrological traceability to the international system of units (SI), which may be to the SI, through a National Metrology Institute such as NIST.

1.2.2. Verify that weight-handling equipment is available and in good operational condition.

[1] This SOP was formerly Good Measurement Practice 14 (2003, 2012).

1.2.3. Verify that the operator is familiar with the design and the operation of the balances and familiar with weighing procedures.

1.3. Safety

1.3.1. Handling of large or small weights can represent a hazard to either the weights or personnel if the weights are dropped.

2. Methodology

2.1. Summary

A sensitivity weight is selected to calibrate the balance over the range to be used in the measurement procedure. Minimizing the difference in mass values between X and S is critical when choosing an appropriate sensitivity weight. Therefore, tare weights may be necessary whenever the difference in mass values is significant. Minimizing the difference between X and S works to our benefit since the range of the measurements is minimized and reduces potential errors that can be introduced by nonlinearity or span inaccuracies of the balance as well as bringing the mass standards within range on mass comparators with limited weighing ranges.

2.2. Apparatus

2.2.1. Clean forceps to handle the weights, or gloves to be worn if the weights are to be moved by hand.

2.3. Procedure for selection

2.3.1. Conduct preliminary measurements to determine the approximate mass value for the difference between the standard and the unknown ($X - S$).

2.3.2. Define the range of use for the balance to be used:

2.3.2.1. Equal arm – number of scale divisions

2.3.2.2. Mechanical – optical scale

2.3.2.3. Combination (Electro-mechanical) and Comparators – digital indications

2.3.2.4. Fully electronic – capacity

2.3.3. Determine the need for tare weights if the difference between *X* and *S* exceeds the values shown in Table 1.

Table 1. Recommended maximum difference between *X* and *S*.

Balance	*(X – S)*
Equal arm	balance each other within one division on the scale
Mechanical	The smaller of 1/10 optical scale or the applicable tolerance
Combination Electro-mechanical and Comparator	The smaller of 1/10 digital range or the applicable tolerance
Fully electronic	The smaller of 0.05 % capacity or the applicable tolerance

2.3.4. Select tare weights, if necessary, making sure that the difference between *X* and *S*, with the appropriate tare weights, do not exceed the values shown in Table 1. If weights are of equal nominal value and within applicable tolerances, the need for tare weights is rare. Tare weights or multiple standards in summation are often required for unequal nominal values.

2.3.5. Select a sensitivity weight within the ranges give according to Table 2. Round the estimated mass of the sensitivity weight to the nearest convenient standard nominal mass (1-2-3-5).

Table 2. Selection of Sensitivity Weight.

Balance	Procedure	Sensitivity Weight
Equal Arm	SOP 3, 5, 6, 7, 8, 28	change turning points by about 20 %
Mechanical	SOP 4, 5, 7, 28 SOP 8	≥ 4 times (*X* – *S*); ≤ ½ optical scale, usually ≈ ¼ optical scale
Combination Electro-mechanical and Comparator	SOP 4, 5, 7, 8, 28	≥ 4 times (*X* – *S*); ≤ ½ digital range
Fully Electronic	SOP 4, 5, 7, 28 SOP 8	≥ 2 times the applicable tolerance ≤ 0.5 % capacity

2.3.5.1. A sensitivity weight is not required if using an electronic mass comparator or fully electronic balance that has been tested (with supporting data and documented analysis available), and which has ongoing periodic validation (e.g., prior to each use)

to determine that the balance has sufficient accuracy, resolution, repeatability, and stability so that no advantage is gained using a sensitivity weight. For example, any possible errors must be less than the last digit retained in the expanded uncertainty. Monitoring is required to verify metrological traceability. See calculations and uncertainties noted in Section 3.

2.6. Use of sensitivity weight

The sensitivity weight is incorporated into the mass procedures to ensure that the mass differences determined with the optical scale, or electronic range, have valid accuracy and traceability. The sensitivity weight calibrates the range of use of the balance used for making the mass determinations. Using a sensitivity weight provides us with a sensitivity value in terms of mass units per division. If the sensitivity is not constant with time, temperature and load, its variation must be included in the mass correction and in the uncertainty. What follows is a generic equation for the sensitivity correction factor. Equations are modified in each SOP when buoyancy corrections are performed.

$$sensitivity = \frac{mass\ units}{divisions} = \frac{M_{sw}}{deflection}$$

where M_{sw} represents the mass of the sensitivity weight.

3. Calculations

3.1. See each mass SOP for calculation of sensitivity as it is included in the procedure. Examples of sensitivity accuracy evaluation include the following:

3.1.1. SOP 8 – The error in sensitivity must be less than 2 percent of the balance reading. That is, the sensitivity factor portion of the mass calculation must be between 0.98 and 1.02 mass units per division when the sensitivity is equal to 1 (or 980 to 1020 if sensitivity is equal to 1000).

3.1.2. Comparison SOPs – The potential systematic error due to sensitivity inaccuracies may be calculated by determining the average observed deflection of a sensitivity weight divided by the mass of the sensitivity weight and multiplied by the average or maximum difference between X and S, from the following equation:

$$Potential\ sensitivity\ error = \left| \frac{\text{Observed deflection - } M_{sw}}{M_{sw}} \right| * \text{Average max } d$$

The applicable tolerance may be substituted for the average maximum difference between X and S, *d*.

4. Uncertainty

4.1. Sensitivity errors that may be incorporated in SOP 8 where sensitivity is assessed but not included in the calculations need to be evaluated and included as an uncorrected systematic error in the uncertainty, according to this approach and to instructions in SOP 8.

4.2. The uncertainty of the sensitivity weight may be treated in the same way as the difference between the observed deflection and mass of the sensitivity weight in the equation given in 3.1.2 to determine significance.

$$Potential\ sensitivity\ uncertainty = \left| \frac{u_{sw}}{M_{sw}} \right| * \text{Average max } d$$

Again, the applicable tolerance may be substituted for the average maximum difference, *d*. The uncertainty of the sensitivity weight is generally relatively small and insignificant. However, it does no harm to incorporate it in uncertainty calculations when spreadsheets are set up to handle all of the data which will account for possible larger uncertainties on sensitivity weights.

4.3. The resulting systematic errors that are calculated in Section 3.1.2 that are evaluated and not corrected as a part of the procedure may be treated as a rectangular distribution in combination with all other sources of uncertainty. Note: An alternative equation such as C.6.4.2, from OIML R111 may be used as well.

4.4. Uncertainties associated with all tare weights are treated as if multiple standards are used in summation and evaluated accordingly (See SOP 29 for references to dependencies).

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8. STATISTICAL TECHNIQUES

Statistics are used in metrology to summarize experimental data, to provide the basis for assessing its quality, and to provide a basis for making probabilistic decisions in its use. The essential basic statistical information for describing a simple data set is:

The mean of the set,	$\overline{x}$
The standard deviation of the set,	s
The number of individuals in the set,	n

If the set is a random sample of the population from which it was derived, if the measurement process is in statistical control, and if all of the observations are independent of one another, then s is an estimate of the population standard deviation, σ, and $\overline{x}$ is an unbiased estimate of the mean, μ.

The population consists of all possible measurements that could have been made under the test conditions for a stable test sample. In this regard, the metrologist must be aware that any changes in the measurement system (known or unknown) could possibly result in significant changes in its operational characteristics, and, hence the values of the mean and standard deviation. Whenever there is doubt, statistical tests should be made to determine the significance of any apparent differences before statistics are combined.

The following discussion reviews some useful statistical techniques for interpreting measurement data. In presenting this information, it is assumed that the reader is already familiar with basic statistical concepts. For a detailed discussion of the following techniques and others not presented here, it is recommended that the reader consult NIST Handbook 91 - Experimental Statistics, by Mary G. Natrella [19]. That handbook also contains comprehensive statistical tables from which the tables contained in Chapter 9 of this publication were taken.

8.1 Estimation of Standard Deviation from a Series of Measurements on a Given Object

Given n measurements $x_1, x_2, x_3, \ldots\ldots, x_n$

Mean, $\overline{x} = \dfrac{(x_1 + x_2 + x_3 + \ldots + x_n)}{n}$

Standard deviation estimate, $s = \sqrt{\dfrac{\sum(x_n - \overline{x})^2}{n-1}}$

The estimate, s, is based on n-1 degrees of freedom

8.2 Estimation of Standard Deviation from the Differences of k Sets of Duplicate Measurements

Given k differences of duplicate measurements, d_1, d_2, d_3, …, d_k, a useful formula for estimating the standard deviation is:

$s_d = \frac{\sum d_i^2}{2k}$ where s_d is based on k degrees of freedom.

Note that $d_1 = \bar{x}_i' - \bar{x}_i''$, for example.

The values d_1, d_2 etc., may be differences of duplicate measurements of the same sample (or object) at various times, or they may be the differences of duplicate measurements of several similar samples (or objects).

8.3 Estimation of Standard Deviation from the Average Range of Several Sets of Measurements

The range, R, is defined as the difference between the largest and smallest values in a set of measurements.

Given R_1, R_2, R_3, …, R_k

Mean, $\bar{R} = \frac{(R_1 + R_2 + R_3 + \ldots + R_k)}{k}$

Standard deviation can be estimated by the formula, $s_R = \frac{\bar{R}}{d_2^*}$

The value of d_2^* will depend on the number of sets of measurements used to calculate s_R, and on the number of measurements in each set, i.e., 2 for duplicates, 3 for triplicates, etc. Consult a table such as Table 9.1 for the appropriate value of d_2^* to use. The effective number of degrees of freedom for s_R is in the table. Note: It is recommended to calculate the standard deviation for each set of replicates and then to calculate a pooled standard deviation whenever possible.

8.4 Pooling Estimates of Standard Deviations

Estimates of the standard deviation obtained at several times may be combined (pooled) to obtain a better estimate based upon more degrees of freedom. The following equation may be used for this purpose:

$$s_p = \sqrt{\frac{(n_1 - 1)s_1^2 + (n_2 - 1)s_2^2 + (n_3 - 1)s_3^2 + \ldots + (n_k - 1)s_k^2}{(n_1 - 1) + (n_2 - 1) + (n_3 - 1) + \ldots + (n_k - 1)}}$$ where

s_p will be based on $(n_1 - 1) + (n_2 - 1) + (n_3 - 1) + \ldots + (n_k - 1)$ degrees of freedom.

8.5 "Within" and "Between" Standard Deviation

Estimation of the within-series, s_w, and between-series, s_b standard deviation, (also referred to as short-term and long-term standard deviations in the applications described here) is an important way to characterize a measurement process. The former provides guidance as to how many repetitions of a measurement are required to obtain a result on a single occasion with a given precision, while the latter is a better estimate of the precision of replication (reproducibility) of a result on various occasions and is a more realistic evaluation of measurement variability.

To estimate these standard deviations, sets of measurements may be made on several occasions. To simplify the calculations, each set should consist of the same number of measurements. For most measurements, it is recommended that duplicate measurements be made on at least 12 separate occasions when estimating s_w and s_b.

Given k sets of duplicate measurements made on k occasions the following table and calculation can be made.

Table 8.1

Occasion	Measured Values		Range	Mean
1	x_1'	x_1''	R_1	$\frac{(x_1' + x_1'')}{2} = \bar{x}_1$
2	x_2'	x_2''	R_2	$\frac{(x_2' + x_2'')}{2} = \bar{x}_2$
3	x_3'	x_3''	R_3	$\frac{(x_3' + x_3'')}{2} = \bar{x}_3$
.	.	.	.	.
.	.	.	.	.
.	.	.	.	.
k	x_k'	x_k''	R_k	$\frac{(x_k' + x_k'')}{2} = \bar{x}_k$

1. Calculate $\bar{R} = \frac{(R_1 + R_2 + R_3 + \ldots + R_k)}{k}$

2. Calculate $s_w = s_R - \frac{\bar{R}}{d_2^*}$

Note: One may use the procedure of 8.2 to calculate s_w if preferred.

3. Calculate s_x as follows:

$$\bar{\bar{x}} = \frac{\left(\bar{x}_1 + \bar{x}_2 + \bar{x}_3 + \ldots + \bar{x}_k\right)}{k}$$

$$s_{\bar{x}} = \sqrt{\frac{\sum\left(\bar{x}_k - \bar{\bar{x}}\right)^2}{k-1}}$$

4. Calculate s_b (for the case of duplicate measurements)

$$s_b = \sqrt{s_x^2 - \frac{s_w^2}{2}}$$

Note that s_b is an estimate of the long term component of the standard deviation of a single measurement. The long term standard deviation of the mean of n measurements taken at a single occasion is estimated by:

$$s_b(\bar{x}_n) = \sqrt{s_b^2 - \frac{s_w^2}{n}}$$

Important note: Do not use this approach for handling within-series and between-time standard deviations with the Mass Code. Separate formulas are available for that application.

8.6 Confidence Interval for the Mean

The estimation of the confidence interval for the mean of n measurements is one of the most frequently used statistical calculations. The formula used will depend on whether the population standard deviation,σ, is known or whether it is estimated on the basis of measurements of a sample(s) of the population.

Using Population Standard Deviation, σ

Strictly speaking, σ, is never known for a measurement process. However, the formula for use in such a case is:

$$\bar{x} \pm \frac{z\sigma}{\sqrt{n}}$$

Variable	Description
$\bar{x}$	sample mean
s	known standard deviation
n	number of measurements of sample
z	standard normal variate, depending on the confidence level desired

For 95 % confidence z = 1.960; for 99.7 % confidence z = 3.0.
For other confidence levels, see Table 9.2

Using Estimate of Standard Deviation, s

In the usual situation, s is known, based on υ degrees of freedom and the formula for use is:

$$\bar{x} \pm \frac{ts}{\sqrt{n}}$$

Variable	Description
$\bar{x}$	sample mean
s	estimate of standard deviation
n	number of measurements on which the mean is based
t	Student's t value, based on the confidence level desired and the υ degrees of freedom associated with s (see Table 9.3).

Note that $t \rightarrow z$ as $n \rightarrow \infty$. For many practical purposes, the standard deviation may be considered as known when estimated by at least 30 degrees of freedom.

8.7 Confidence Interval for σ

The standard deviation, σ, is ordinarily not known but is, instead, an estimated value based on a limited number of measurements, using procedures such as have been described above. Such estimates may be pooled, as appropriate, to obtain better estimates. In any case, the uncertainty of the estimated value of the standard deviation may be of interest and can be expressed in the form of a confidence interval, computed as indicated below.

The interval is asymmetrical because the standard deviation is ordinarily underestimated when small numbers of measurements are involved due to the fact that large deviations occur infrequently in a limited measurement process. Indeed, it is the general experience of metrologists that a few measurements appear to be more precise than they really are.

The basic information required to compute the interval is an estimate of the standard deviation, s, and the number of degrees of freedom on which the estimate is based. The relationships to use are:

Lower limit $B_L s$

Upper limit $B_U s$

Interval $B_L s$ to $B_U s$

The values for B_L and B_U depend upon the confidence level and degrees of freedom associated with s. Values for use in calculating the confidence level are given in Table 9.7. A more extensive table (Table A-20) is available in NIST Handbook 91 [19].

8.8 Statistical Tolerance Intervals

Statistical tolerance intervals define the bounds within which a percentage of the population is expected to lie with a given level of confidence. For example, one may wish to define the limits within which 95 % of measurements would be expected to lie with a 95 % confidence of being correct. The interval is symmetrical and is computed using the expression

$$\bar{x} \pm ks$$

where k depends on three things

Variable	Description
p	the proportion or percentage of the individual measurements to be included
γ	the confidence coefficient to be associated with the interval
n	the number of measurements on which the estimate, s, is based

Table 9.6 may be used to obtain values for k for frequently desired values of γ and p. A more extensive table is Table A-6 found in NIST Handbook 91 [19].

8.9 Comparing Estimates of a Standard Deviation (F-Test)

The F-test may be used to decide whether there is sufficient reason to believe that two estimates of a standard deviation differ significantly. The ratio of the variances (square of the standard deviation) is calculated and compared with tabulated values. Unless the computed ratio is larger than the tabulated value, there is no reason to believe that the observed standard deviations are significantly different.

The F-ratio is calculated using the equation

$$F = \frac{s_L^2}{s_S^2}$$

where s_L is the numerically larger value and s_S is the smaller value of the two estimates under consideration.

The critical value of F depends on the significance level chosen for the decision (test) and the number of degrees of freedom associated with s_L and s_S, respectively.

Table 9.4 contains critical values for F at the 95 % level of confidence. The tabulated values of F are not expected to be exceeded with 95 % confidence on the basis of chance alone. As an example, if both the numerator and the denominator values for s were each based on 9 degrees of freedom, an F value no larger than 4.03 is expected with 95 % confidence, due to the uncertainties of the s values, themselves. Table A-5 of NIST Handbook 91 [19] contains values for F for other confidence levels.

The F-test is useful for comparing the precision of methods, equipment, laboratories, or metrologists, for example. An inspection of Table 9.4 shows that when either of the values of *s* is based on a small number of degrees of freedom, the F value is large. Consequently, the significance of decisions based on small changes in precision can be supported statistically only by a relatively large number of measurements. If such changes are suspected, but the data requirement is difficult to meet, the decision may need to be made on the basis of information about the measurement process itself.

The F-test is also useful for deciding whether estimates of the standard deviation made at various times differ significantly. Such questions need to be answered when deciding on whether to revise control limits of a control chart, for example.

8.10 Comparing a Set of Measurements with a Given Value

The question may arise as to whether a measured value agrees or significantly disagrees with a stated value for the measured object. The evaluation can be based on whether or not the confidence interval for the measured value encompasses the stated value. The confidence interval is calculated using the expression

$$\bar{x} \pm \frac{ts}{\sqrt{n}}$$

as previously described in Section 8.6. In using this expression, *n* represents the number of measurements used to calculate the mean, $\bar{x}$, and *t* depends on the degrees of freedom, υ, associated with *s* and the confidence level needed when making the decision. Note that one may use historical data for estimating *s*, such as a control chart for example, in which case υ will represent the degrees of freedom associated with establishment of the control limits and may be considerably larger than $n-1$.

8.11 Comparing Two Sets of Measurements with Regard to Their Means

This discussion is concerned with deciding whether the means of two measured values, $_A$ and $_B$, are in agreement. The data sets used for this purpose may consist of the following:

$\bar{x}_A$	$\bar{x}_B$
s_A	s_B
n_A	n_B

The first question to be resolved is whether s_A and s_B can be considered to be different estimates of the same standard deviation or whether they do, indeed, differ. An F test may be used for this purpose. However, it will be recalled that this is not sensitive to small real differences, so the decision may need to be based on physical considerations, such as the known stability of the measurement process, for example.

Case I

Confirming (or assuming) that s_A and s_B are not significantly different, they are pooled, as already described (but repeated here for convenience) and used to calculate a confidence interval for the difference of the means. If this is larger than the observed difference, there is no reason to believe that the means differ. The steps to follow when making the calculation described above are:

Step 1. Choose α, the level of significance for the test

Step 2. Calculate the pooled estimate of the standard deviation, s_p

$$s_p = \sqrt{\frac{(n_A - 1)s_A^2 + (n_B - 1)s_B^2}{(n_A - 1) + (n_B - 1)}}$$

s_p will be estimated with $n_A + n_B - 2$ degrees of freedom

Step 3. Calculate the respective variances of the means

$$v_A = \frac{s_A^2}{n_A} \text{ and } v_B = \frac{s_B^2}{n_B}$$

Step 4. Calculate the uncertainty of $|X_A - X_B| = \Delta$

$$U_\Delta = t\sqrt{(V_A + V_B)}$$

using a value for t based on $\frac{\alpha}{2}$ and $\upsilon = n_A + n_B - 2$.

Step 5. Compare U_Δ with Δ

If $U_\Delta \geq \Delta$, there is no reason to believe that Δ is significant at the level of confidence chosen.

Case II

Confirming (or assuming) that s_A and s_B are significantly different, their individual values are used to calculate U_Δ as outlined below.

Step 1. Choose α, the level of significance for the test.

Step 2. Calculate the respective variances of the means.

$$v_A = \frac{s_A^2}{n_A} \text{ and } v_B = \frac{s_B^2}{n_B}$$

Step 3. Calculate the uncertainty of $|X_A - X_B| = \Delta$

$$U_{\Delta} = t\sqrt{(\nu_A + \nu_B)}$$

using a value for t based on $\frac{\alpha}{2}$ and f, the effective number of degrees of freedom calculated as described in Step 4.

Step 4. Calculate f, the effective number of degrees of freedom using the equation below or the Welch-Satterthwaite formula given in NIST Technical Note 1297 or the Guide to the Expression of Uncertainty in Measurement:

$$f = \left(\frac{\left(V_A^2 + V_B^2\right)}{\frac{V_A^2}{f_A} + \frac{V_B^2}{f_B}} \right)$$

Step 5. Compare U_{Δ} with Δ.

If $U_{\Delta} \geq \Delta$, there is no reason to believe that Δ is significant at the level of confidence chosen.

8.12 Use of Random Numbers

Conducting operations in random sequences can avert problems of bias that might stem from a particular order of operations. For example, in the measurement of a series of items, it might be difficult to determine whether systematic trends in the measured values were due to differences in the items or to measurement system drift unless the items were measured in random order.

Use of tables of random numbers is a convenient means for randomizing measurement operations. The operations, test objects, and other matters requiring randomization may be assigned serial numbers. The order of selection is then determined by use of a random number table, as described below. When the number of operations or test items is less than 100, a table such as Table 9.11, reproduced from NIST Handbook 91 [19], may be used conveniently. One may start from any arbitrarily selected position in the table and proceed from it in any pre-determined arbitrary manner. If the first number encountered is not that of one of any item, ignore it and proceed until a valid match is encountered. This becomes the first item in the sequence. Continuing in the same manner, items are selected in the sequence in which their serial numbers are encountered ignoring the repetition of previously identified items. The procedure is continued until all items have been randomly selected.

As an example, select 10 specimens (numbered 01 to 10) in random order. Start from a randomly selected place, say column 2, row 5 of Table 9.11. Proceed from this point along the table as one would read a book. The starting number is 14, which is not usable. The first useful number encountered is 08, the next 03, and so on. Using the procedure described above, the following random order was found:

Specimen No.									
08	03	09	05	06	02	07	10	04	01
Order									
1	2	3	4	5	6	7	8	9	10

9. Reference Tables

Table 9.1 Use of Range to Estimate Standard Deviation

Table 9.2 Z Factors for Two-sided Confidence Intervals

Table 9.3 Student t-Variate

Table 9.4 Critical Values for the F-test, $F_{0.975}$

Table 9.5 Critical Values for the F-test, $F_{0.99}$

Table 9.6 Two-sided Tolerance Interval Factors

Table 9.7 Factors for Computing Two-sided Confidence Intervals for

Table 9.8 Density of Air-free Water

Table 9.9 Density of Air

Table 9.10 Coefficient of Linear and Cubical Expansion for Materials Commonly Used in Laboratory Apparatus and Standards

Table 9.11 A Short Table of Random Numbers

Table 9.1 Use of Range to Estimate Standard Deviation

Number of Sets of Replicates k	Factor Degrees of Freedom	Number of Replicates in a Set				
		2	3	4	5	6
1	d_2^* υ	1.41 1.00	1.91 1.98	2.24 2.93	2.48 3.83	2.67 4.68
3	d_2^* υ	1.23 2.83	1.77 5.86	2.12 8.44	2.38 11.1	2.58 13.6
5	d_2^* υ	1.19 4.59	1.74 9.31	2.10 13.9	2.36 18.4	2.56 22.6
10	d_2^* υ	1.16 8.99	1.72 18.4	2.08 27.6	2.34 36.5	2.55 44.9
15	d_2^* υ	1.15 13.4	1.71 27.5	2.07 41.3	2.34 54.6	2.54 67.2
20	d_2^* υ	1.14 17.8	1.70 36.5	2.07 55.0	2.33 72.7	2.54 89.6
∞	d_2^*	1.13	1.69	2.06	2.33	2.53
$s_p = \frac{\overline{R}}{d_2^*}$						

Intermediate values for d_2^* and ν may be obtained by interpolation, or from the reference from which this table was adapted. Example: When using a Range chart for volume calibrations, based on two runs for each unknown test item, the d_2^* value is 1.41. If testing 10 points on a tape measure, using 3 runs, the average range value is calculated for all points and d_2^* is 1.72.

Adapted from Lloyd S. Nelson, J. Qual. Tech. 7 No. 1, January 1975. ©American Society for Quality Control, Reprinted by permission.

Table 9.2 Z-Factors for Two-sided Confidence Intervals for the Normal Distribution

Confidence Level. %	Z Factor
50	0.68
67	1.00
75	1.15
90	1.645
95	1.960
95.28	2.000
99.00	2.575
99.74	3
99.993 4	4
99.999 95	5
100, 10^{-9}	6
100, 10^{-12}	7
100, 10^{-15}	8
100, $10^{-18.9}$	9
100, 10^{-23}	10

Table 9.3 Student t-Variate

*	80%	90%	95%	98%	99%	99.73%
df	$t_{0.90}$	$t_{0.95}$	$t_{0.975}$	$t_{0.99}$	$t_{0.995}$	$t_{0.9985}$
1	3.078	6.314	12.706	31.821	63.657	235.80
2	1.886	2.920	4.303	6.965	9.925	19.207
3	1.638	2.353	3.182	4.541	5.841	9.219
4	1.533	2.132	2.776	3.747	4.604	6.620
5	1.476	2.015	2.571	3.365	4.032	5.507
6	1.440	1.943	2.447	3.143	3.707	4.904
7	1.415	1.895	2.365	2.998	3.499	4.530
8	1.397	1.860	2.306	2.896	3.355	4.277
9	1.383	1.833	2.262	2.821	3.250	4.094
10	1.372	1.812	2.228	2.764	3.169	3.957
11	1.363	1.796	2.201	2.718	3.106	3.850
12	1.356	1.782	2.179	2.681	3.055	3.764
13	1.350	1.771	2.160	2.650	3.012	3.694
14	1.345	1.761	2.145	2.624	2.977	3.636
15	1.341	1.753	2.131	2.602	2.947	3.586
16	1.337	1.746	2.120	2.583	2.921	3.544
17	1.333	1.740	2.110	2.567	2.898	3.507
18	1.330	1.734	2.101	2.552	2.878	3.475
19	1.328	1.729	2.093	2.539	2.861	3.447
20	1.325	1.725	2.086	2.528	2.845	3.422
25	1.316	1.708	2.060	2.485	2.787	3.330
30	1.310	1.697	2.042	2.457	2.750	3.270
40	1.303	1.684	2.021	2.423	2.704	3.199
60	1.296	1.671	2.000	2.390	2.660	3.130
∞	1.282	1.645	1.960	2.326	2.576	3.00

* Columns to be used in calculating corresponding two-sided confidence interval. From: NIST Handbook 91 p. T-5; Last column from B.J. Joiner, J. Research NIST.

Table 9.4 Critical values for the F-Test, $F_{0.975}$

n_1 = degrees of freedom for numerator n_2 = degrees of freedom for denominator

n_2 \ n_1	1	2	3	4	5	6	7	8	9	10	12	15	20	24	30	40
1	647.8	799.5	864.2	899.6	921.8	937.1	948.2	956.7	963.3	968.6	976.7	984.9	993.1	997.2	1001	1006
2	38.51	39.00	39.17	39.25	39.30	39.33	39.36	39.37	39.39	39.40	39.41	39.43	39.45	39.46	39.46	39.47
3	17.44	16.04	15.44	15.10	14.88	14.73	14.62	14.54	14.47	14.42	14.34	14.25	14.17	14.12	14.08	14.04
4	12.22	10.65	9.98	9.60	9.36	9.20	9.07	8.98	8.90	8.84	8.75	8.66	8.56	8.51	8.46	8.41
5	10.01	8.43	7.76	7.39	7.15	6.98	6.85	6.76	6.68	6.62	6.52	6.43	6.33	6.28	6.23	6.18
6	8.81	7.26	6.60	6.23	5.99	5.83	5.70	5.60	5.52	5.46	5.37	5.27	5.17	5.12	5.07	5.01
7	8.07	6.54	5.89	5.52	5.29	5.12	4.99	4.90	4.82	4.76	4.67	4.57	4.47	4.42	4.36	4.31
8	7.57	6.06	5.42	5.05	4.82	4.65	4.53	4.43	4.36	4.30	4.20	4.10	4.00	3.95	3.89	3.84
9	7.21	5.71	5.08	4.72	4.48	4.32	4.20	4.10	4.03	3.96	3.87	3.77	3.67	3.61	3.56	3.51
10	6.94	5.46	4.83	4.47	4.24	4.07	3.95	3.85	3.78	3.72	3.62	3.52	3.42	3.37	3.31	3.26
11	6.72	5.26	4.63	4.28	4.04	3.88	3.76	3.66	3.59	3.53	3.43	3.33	3.23	3.17	3.12	3.06
12	6.55	5.10	4.47	4.12	3.89	3.73	3.61	3.51	3.44	3.37	3.28	3.18	3.07	3.02	2.96	2.91
13	6.41	4.97	4.35	4.00	3.77	3.60	3.48	3.39	3.31	3.25	3.15	3.05	2.95	2.89	2.84	2.78
14	6.30	4.86	4.24	3.89	3.66	3.50	3.38	3.29	3.21	3.15	3.05	2.95	2.84	2.79	2.73	2.67
15	6.20	4.77	4.15	3.80	3.58	3.41	3.29	3.20	3.12	3.06	2.96	2.86	2.76	2.70	2.64	2.59
16	6.12	4.69	4.08	3.73	3.50	3.34	3.22	3.12	3.05	2.99	2.89	2.79	2.68	2.63	2.57	2.51
17	6.04	4.62	4.01	3.66	3.44	3.28	3.16	3.06	2.98	2.92	2.82	2.72	2.62	2.56	2.50	2.44
18	5.98	4.56	3.95	3.61	3.38	3.22	3.10	3.01	2.93	2.87	2.77	2.67	2.56	2.50	2.44	2.38
19	5.92	4.51	3.90	3.56	3.33	3.17	3.05	2.96	2.88	2.82	2.72	2.62	2.51	2.45	2.39	2.33
20	5.87	4.46	3.86	3.51	3.29	3.13	3.01	2.91	2.84	2.77	2.68	2.57	2.46	2.41	2.35	2.29
21	5.83	4.42	3.82	3.48	3.25	3.09	2.97	2.87	2.80	2.73	2.64	2.53	2.42	2.37	2.31	2.25
22	5.79	4.38	3.78	3.44	3.22	3.05	2.93	2.84	2.76	2.70	2.60	2.50	2.39	2.33	2.27	2.21
23	5.75	4.35	3.75	3.41	3.18	3.02	2.90	2.81	2.73	2.67	2.57	2.47	2.36	2.30	2.24	2.18
24	5.72	4.32	3.72	3.38	3.15	2.99	2.87	2.78	2.70	2.64	2.54	2.44	2.33	2.27	2.21	2.15
25	5.69	4.29	3.69	3.35	3.13	2.97	2.85	2.75	2.68	2.61	2.51	2.41	2.30	2.24	2.18	2.12
26	5.66	4.27	3.67	3.33	3.10	2.94	2.82	2.73	2.65	2.59	2.49	2.39	2.28	2.22	2.16	2.09
27	5.63	4.24	3.65	3.31	3.08	2.92	2.80	2.71	2.63	2.57	2.47	2.36	2.25	2.19	2.13	2.07
28	5.61	4.22	3.63	3.29	3.06	2.90	2.78	2.69	2.61	2.55	2.45	2.34	2.23	2.17	2.11	2.05
29	5.59	4.20	3.61	3.27	3.04	2.88	2.76	2.67	2.59	2.53	2.43	2.32	2.21	2.15	2.09	2.03
30	5.57	4.18	3.59	3.25	3.03	2.87	2.75	2.65	2.57	2.51	2.41	2.31	2.20	2.14	2.07	2.01
40	5.42	4.05	3.46	3.13	2.90	2.74	2.62	2.53	2.45	2.39	2.29	2.18	2.07	2.01	1.94	1.88
60	5.29	3.93	3.34	3.01	2.79	2.63	2.51	2.41	2.33	2.27	2.17	2.06	1.94	1.88	1.82	1.74
120	5.15	3.80	3.23	2.89	2.67	2.52	2.39	2.30	2.22	2.16	2.05	1.94	1.82	1.76	1.69	1.61
∞	5.02	3.69	3.12	2.79	2.57	2.41	2.29	2.19	2.11	2.05	1.94	1.83	1.71	1.64	1.57	1.48

For use for a one-tailed test of equality of standard deviation estimate at 2.5 % level of confidence, or for a two-tailed test at 5 % level of confidence.

Table 9.5 Critical values for the F-Test, $F_{0.99}$

υ_1 = degrees of freedom for numerator υ_2 = degrees of freedom for denominator

υ_2 \ υ_1	1	2	3	4	5	6	7	8	9	10
10	10.04	7.56	6.55	5.99	5.64	5.39	5.20	5.06	4.94	4.85
11	9.65	7.21	6.22	5.67	5.32	5.07	4.89	4.74	4.63	4.54
12	9.33	6.93	5.95	5.41	5.06	4.82	4.64	4.50	4.39	4.30
13	9.07	6.70	5.74	5.21	4.86	4.62	4.44	4.30	4.19	4.10
14	8.86	6.51	5.56	5.04	4.69	4.46	4.28	4.14	4.03	3.94
15	8.68	6.36	5.42	4.89	4.56	4.32	4.14	4.00	3.89	3.80
16	8.53	6.23	5.29	4.77	4.44	4.20	4.03	3.89	3.78	3.69
17	8.40	6.11	5.18	4.67	4.34	4.10	3.93	3.79	3.68	3.59
18	8.29	6.01	5.09	4.58	4.25	4.01	3.84	3.71	3.60	3.51
19	8.18	5.93	5.01	4.50	4.17	3.94	3.77	3.63	3.52	3.43
20	8.10	5.85	4.94	4.43	4.10	3.87	3.70	3.56	3.46	3.37
22	7.95	5.72	4.82	4.31	3.99	3.76	3.59	3.45	3.35	3.26
24	7.82	5.61	4.72	4.22	3.90	3.67	3.50	3.36	3.26	3.17
26	7.72	5.53	4.64	4.14	3.82	3.59	3.42	3.29	3.18	3.09
28	7.64	5.45	4.57	4.07	3.75	3.53	3.36	3.23	3.12	3.03
30	7.56	5.39	4.51	4.02	3.70	3.47	3.30	3.17	3.07	2.98
35	7.42	5.27	4.40	3.91	3.59	3.37	3.20	3.07	2.96	2.88
40	7.31	5.18	4.31	3.83	3.51	3.29	3.12	2.99	2.89	2.80
45	7.23	5.11	4.25	3.77	3.45	3.23	3.07	2.94	2.83	2.74
50	7.17	5.06	4.20	3.72	3.41	3.19	3.02	2.89	2.78	2.70
55	7.12	5.01	4.16	3.68	3.37	3.15	2.98	2.85	2.75	2.66
60	7.08	4.98	4.13	3.65	3.34	3.12	2.95	2.82	2.72	2.63
65	7.04	4.95	4.10	3.62	3.31	3.09	2.93	2.80	2.69	2.61
70	7.01	4.92	4.07	3.60	3.29	3.07	2.91	2.78	2.67	2.59
75	6.99	4.90	4.05	3.58	3.27	3.05	2.89	2.76	2.65	2.57
80	6.96	4.88	4.04	3.56	3.25	3.04	2.87	2.74	2.64	2.55
85	6.94	4.86	4.02	3.55	3.24	3.02	2.86	2.73	2.62	2.54
90	6.93	4.85	4.01	3.53	3.23	3.01	2.84	2.72	2.61	2.52
95	6.91	4.84	3.99	3.52	3.22	3.00	2.83	2.70	2.60	2.51
100	6.90	4.82	3.98	3.51	3.21	2.99	2.82	2.69	2.59	2.50
105	6.88	4.81	3.97	3.50	3.20	2.98	2.81	2.69	2.58	2.49
110	6.87	4.80	3.96	3.49	3.19	2.97	2.81	2.68	2.57	2.49
115	6.86	4.79	3.96	3.49	3.18	2.96	2.80	2.67	2.57	2.48
120	6.85	4.79	3.95	3.48	3.17	2.96	2.79	2.66	2.56	2.47
∞	6.63	4.61	3.78	3.32	3.02	2.80	2.64	2.51	2.41	2.32

For use for a one-tailed test of equality of standard deviation estimate at 1 % level of confidence, or for a two-tailed test at 2 % level of confidence.

Table 9.6 Factors for Two-sided Tolerance Intervals for the Normal Distribution

	= 0.95					= 0.99				
P / n	0.75	0.90	0.95	0.99	0.999	0.75	0.90	0.95	0.99	0.999
2	22.858	32.019	37.674	48.430	60.573	114.363	160.193	188.491	242.300	303.054
3	5.922	8.380	9.916	12.861	16.208	13.378	18.930	22.401	29.055	36.616
4	3.779	5.369	6.370	8.299	10.502	6.614	9.398	11.150	14.527	18.383
5	3.002	4.275	5.079	6.634	8.415	4.643	6.612	7.855	10.260	13.015
6	2.604	3.712	4.414	5.775	7.337	3.743	5.337	6.345	8.301	10.548
7	2.361	3.369	4.007	5.248	6.676	3.233	4.613	5.488	7.187	9.142
8	2.197	3.136	3.732	4.891	6.226	2.905	4.147	4.936	6.468	8.234
9	2.078	2.967	3.532	4.631	5.899	2.677	3.822	4.550	5.966	7.600
10	1.987	2.839	3.379	4.433	5.649	2.508	3.582	4.265	5.594	7.129
11	1.916	2.737	3.259	4.277	5.452	2.378	3.397	4.045	5.308	6.766
12	1.858	2.655	3.162	4.150	5.291	2.274	3.250	3.870	5.079	6.477
13	1.810	2.587	3.081	4.044	5.158	2.190	3.130	3.727	4.893	6.240
14	1.770	2.529	3.012	3.955	5.045	2.120	3.029	3.608	4.737	6.043
15	1.735	2.480	2.954	3.878	4.949	2.060	2.945	3.507	4.605	5.876
16	1.705	2.437	2.903	3.812	4.865	2.009	2.872	3.421	4.492	5.732
17	1.679	2.400	2.858	3.754	4.791	1.965	2.808	3.345	4.393	5.607
18	1.655	2.366	2.819	3.702	4.725	1.926	2.753	3.279	4.307	5.497
19	1.635	2.337	2.784	3.656	4.667	1.891	2.703	3.221	4.230	5.399
20	1.616	2.310	2.752	3.612	4.614	1.860	2.659	3.168	4.161	5.312
21	1.599	2.286	2.723	3.577	4.567	1.833	2.620	3.121	4.100	5.234
22	1.584	2.264	2.697	3.543	4.523	1.808	2.584	3.078	4.044	5.163
23	1.570	2.244	2.673	3.512	4.484	1.785	2.551	3.040	3.993	5.098
24	1.557	2.225	2.651	3.483	4.447	1.764	2.522	3.004	3.947	5.039
25	1.545	2.208	2.631	3.457	4.413	1.745	2.494	2.972	3.904	4.985
26	1.534	2.193	2.612	3.432	4.382	1.727	2.469	2.941	3.865	4.935
27	1.523	2.178	2.595	3.409	4.353	1.711	2.446	2.914	3.828	4.888

From: NIST Handbook 91, p T-11

Table 9.7 Factors for Computing Two-sided Confidence Limits for α (Part 1)

Degrees of Freedom	α = 0.05		α = 0.01		α = 0.001	
	S_U	S_L	S_U	S_L	S_U	S_L
1	17.79	0.3576	86.31	0.2969	844.4	0.2480
2	4.859	0.4581	10.70	0.3879	33.29	0.3291
3	3.183	0.5178	5.449	0.4453	11.65	0.3824
4	2.567	0.5590	3.892	0.4865	6.938	0.4218
5	2.248	0.5899	3.175	0.5182	5.085	0.4529
6	2.052	0.6143	2.764	0.5437	4.128	0.4784
7	1.918	0.6344	2.498	0.5650	3.551	0.5000
8	1.820	0.6513	2.311	0.5830	3.167	0.5186
9	1.746	0.6657	2.173	0.5987	2.894	0.5348
10	1.686	0.6784	2.065	0.6125	2.689	0.5492
11	1.638	0.6896	1.980	0.6248	2.530	0.5621
12	1.598	0.6995	1.909	0.6358	2.402	0.5738
13	1.564	0.7084	1.851	0.6458	2.298	0.5845
14	1.534	0.7166	1.801	0.6549	2.210	0.5942
15	1.509	0.7240	1.758	0.6632	2.136	0.6032
16	1.486	0.7808	1.721	0.6710	2.073	0.6116
17	1.466	0.7372	1.688	0.6781	2.017	0.6193
18	1.448	0.7430	1.658	0.6848	1.968	0.6266
19	1.432	0.7484	1.632	0.6909	1.925	0.6333
20	1.417	0.7535	1.609	0.6968	1.886	0.6397
21	1.404	0.7582	1.587	0.7022	1.851	0.6457
22	1.391	0.7627	1.568	0.7074	1.820	0.6514
23	1.380	0.7669	1.550	0.7122	1.791	0.6568
24	1.370	0.7709	1.533	0.7169	1.765	0.6619
25	1.360	0.7747	1.518	0.7212	1.741	0.6668

From: NIST Handbook 91, p T-34

Table 9.7 Factors for Computing Two-sided Confidence Limits for α (Part 2)

Degrees of Freedom	α = 0.05		α = 0.01		α = 0.001	
	S_U	S_L	S_U	S_L	S_U	S_L
26	1.351	0.7783	1.504	0.7253	1.719	0.7130
27	1.343	0.7817	1.491	0.7293	1.698	0.6758
28	1.335	0.7849	1.479	0.7331	1.679	0.6800
29	1.327	0.7880	1.467	0.7367	1.661	0.6841
30	1.321	0.7909	1.457	0.7401	1.645	0.6880
31	1.314	0.7937	1.447	0.7434	1.629	0.6917
32	1.308	0.7964	1.437	0.7467	1.615	0.6953
33	1.302	0.7990	1.428	0.7497	1.601	0.6987
34	1.296	0.8015	1.420	0.7526	1.588	0.7020
35	1.291	0.8089	1.412	0.7554	1.576	0.7052
36	1.286	0.8062	1.404	0.7582	1.564	0.7083
37	1.281	0.8085	1.397	0.7608	1.553	0.7113
38	1.277	0.8106	1.390	0.7633	1.543	0.7141
39	1.272	0.8126	1.383	0.7658	1.533	0.7169
40	1.268	0.8146	1.377	0.7681	1.523	0.7197
41	1.264	0.8166	1.371	0.7705	1.515	0.7223
42	1.260	0.8184	1.365	0.7727	1.506	0.7248
43	1.257	0.8202	1.360	0.7748	1.498	0.7273
44	1.253	0.8220	1.355	0.7769	1.490	0.7297
45	1.249	0.8237	1.349	0.7789	1.482	0.7320
46	1.246	0.8253	1.345	0.7809	1.475	0.7342
47	1.243	0.8269	1.340	0.7828	1.468	0.7364
48	1.240	0.8285	1.335	0.7847	1.462	0.7386
49	1.237	0.8300	1.331	0.7864	1.455	0.7407
50	1.234	0.8314	1.327	0.7882	1.449	0.7427

From: NIST Handbook 91, p. T-34

Table 9.8 Density of Air-free Water in g/cm^3 as a Function of Celsius Temperature

T	0.0 °C	0.1 °C	0.2 °C	0.3 °C	0.4 °C	0.5 °C	0.6 °C	0.7 °C	0.8 °C	0.9 °C
0.0	0.999 840	0.999 846	0.999 853	0.999 859	0.999 865	0.999 871	0.999 877	0.999 883	0.999 888	0.999 893
1.0	0.999 899	0.999 903	0.999 908	0.999 913	0.999 917	0.999 921	0.999 925	0.999 929	0.999 933	0.999 937
2.0	0.999 940	0.999 943	0.999 946	0.999 949	0.999 952	0.999 954	0.999 956	0.999 959	0.999 961	0.999 963
3.0	0.999 964	0.999 966	0.999 967	0.999 968	0.999 969	0.999 970	0.999 971	0.999 971	0.999 972	0.999 972
4.0	0.999 972	0.999 972	0.999 972	0.999 971	0.999 971	0.999 970	0.999 969	0.999 968	0.999 967	0.999 965
5.0	0.999 964	0.999 962	0.999 960	0.999 958	0.999 956	0.999 954	0.999 951	0.999 949	0.999 946	0.999 943
6.0	0.999 940	0.999 937	0.999 933	0.999 930	0.999 926	0.999 922	0.999 918	0.999 914	0.999 910	0.999 906
7.0	0.999 901	0.999 896	0.999 892	0.999 887	0.999 881	0.999 876	0.999 871	0.999 865	0.999 860	0.999 854
8.0	0.999 848	0.999 842	0.999 835	0.999 829	0.999 822	0.999 816	0.999 809	0.999 802	0.999 795	0.999 787
9.0	0.999 780	0.999 773	0.999 765	0.999 757	0.999 749	0.999 741	0.999 733	0.999 725	0.999 716	0.999 707
10.0	0.999 699	0.999 690	0.999 681	0.999 672	0.999 662	0.999 653	0.999 643	0.999 634	0.999 624	0.999 614
11.0	0.999 604	0.999 594	0.999 583	0.999 573	0.999 562	0.999 552	0.999 541	0.999 530	0.999 519	0.999 507
12.0	0.999 496	0.999 485	0.999 473	0.999 461	0.999 449	0.999 437	0.999 425	0.999 413	0.999 401	0.999 388
13.0	0.999 376	0.999 363	0.999 350	0.999 337	0.999 324	0.999 311	0.999 297	0.999 284	0.999 270	0.999 256
14.0	0.999 243	0.999 229	0.999 215	0.999 200	0.999 186	0.999 172	0.999 157	0.999 142	0.999 128	0.999 113
15.0	0.999 098	0.999 083	0.999 067	0.999 052	0.999 036	0.999 021	0.999 005	0.998 989	0.998 973	0.998 957
16.0	0.998 941	0.998 925	0.998 908	0.998 892	0.998 875	0.998 858	0.998 841	0.998 824	0.998 807	0.997 890
17.0	0.998 773	0.998 755	0.998 738	0.998 720	0.998 702	0.998 684	0.998 666	0.998 648	0.998 630	0.998 612
18.0	0.998 593	0.998 575	0.998 556	0.998 537	0.998 519	0.998 500	0.998 480	0.998 461	0.998 442	0.998 422
19.0	0.998 403	0.998 383	0.998 364	0.998 344	0.998 324	0.998 304	0.998 284	0.998 263	0.998 243	0.998 222
20.0	0.998 202	0.998 181	0.998 160	0.998 139	0.998 118	0.998 097	0.998 076	0.998 055	0.998 033	0.998 012
21.0	0.997 990	0.997 968	0.997 947	0.997 925	0.997 903	0.997 881	0.997 858	0.997 836	0.997 814	0.997 791
22.0	0.997 768	0.997 746	0.997 723	0.997 700	0.997 677	0.997 654	0.997 630	0.997 607	0.997 584	0.997 560
23.0	0.997 536	0.997 513	0.997 489	0.997 465	0.997 441	0.997 417	0.997 392	0.997 368	0.997 344	0.997 319
24.0	0.997 294	0.997 270	0.997 245	0.997 220	0.997 195	0.997 170	0.997 145	0.997 119	0.997 094	0.997 068
25.0	0.997 043	0.997 017	0.996 991	0.996 966	0.996 940	0.996 913	0.996 887	0.996 861	0.996 835	0.996 808
26.0	0.996 782	0.996 755	0.996 728	0.996 702	0.996 675	0.996 648	0.996 621	0.996 593	0.996 566	0.996 539
27.0	0.996 511	0.996 484	0.996 456	0.996 428	0.996 401	0.996 373	0.996 345	0.996 316	0.996 288	0.996 260
28.0	0.996 232	0.996 203	0.996 175	0.996 146	0.996 117	0.996 088	0.996 060	0.996 031	0.996 001	0.995 972
29.0	0.995 943	0.995 914	0.995 884	0.995 855	0.995 825	0.995 795	0.995 765	0.995 736	0.995 706	0.995 676
30.0	0.995 645	0.995 615	0.995 585	0.995 554	0.995 524	0.995 493	0.995 463	0.995 432	0.995 401	0.995 370
31.0	0.995 339	0.995 308	0.995 277	0.995 246	0.995 214	0.995 183	0.995 151	0.995 120	0.995 088	0.995 056
32.0	0.995 024	0.994 992	0.994 960	0.994 928	0.994 896	0.994 864	0.994 831	0.994 799	0.994 766	0.994 734
33.0	0.994 701	0.994 668	0.994 635	0.994 602	0.994 569	0.994 536	0.994 503	0.994 470	0.994 436	0.994 403
34.0	0.994 369	0.994 336	0.994 302	0.994 268	0.994 234	0.994 201	0.994 167	0.994 132	0.994 098	0.994 064
35.0	0.994 030	0.993 995	0.993 961	0.993 926	0.993 891	0.993 857	0.993 822	0.993 787	0.993 752	0.993 717
36.0	0.993 682	0.993 647	0.993 611	0.993 576	0.993 541	0.993 505	0.993 469	0.993 434	0.993 398	0.993 362
37.0	0.993 326	0.993 290	0.993 254	0.993 218	0.993 182	0.993 146	0.993 109	0.993 073	0.993 036	0.993 000
38.0	0.992 963	0.992 926	0.992 889	0.992 852	0.992 815	0.992 778	0.992 741	0.992 704	0.992 667	0.992 629
39.0	0.992 592	0.992 554	0.992 517	0.992 479	0.992 442	0.992 404	0.992 366	0.992 328	0.992 290	0.992 252

*Based on the work of H. Wagenbreth and W. Blanke, PTB - Mitteilingen 6-71.
Reference equations for Air-Saturated distilled water should be used and are provided in GLP 10 as published in NISTIR 7383, see latest edition.

Table 9.9 Density of Air, g/cm^3, as Function of Temperature and Air Pressure*

Barometric Pressure			Temperature, °C					
kPa	**mbar**	**mm Hg**	**18**	**20**	**22**	**24**	**26**	**28**
77.33	773	580	0.000 92	0.000 92	0.000 91	0.000 91	0.000 90	0.000 90
79.99	800	600	0.000 95	0.000 95	0.000 94	0.000 93	0.000 93	0.000 92
82.66	827	620	0.000 99	0.000 98	0.000 97	0.000 96	0.000 96	0.000 95
85.33	853	640	0.001 02	0.001 01	0.001 00	0.001 00	0.000 99	0.000 98
87.99	880	660	0.001 05	0.001 04	0.001 03	0.001 03	0.001 02	0.001 01
90.66	907	680	0.001 08	0.001 07	0.001 07	0.001 06	0.001 05	0.001 04
93.33	933	700	0.001 11	0.001 10	0.001 10	0.001 09	0.001 08	0.001 07
95.99	960	720	0.001 14	0.001 14	0.001 13	0.001 12	0.001 12	0.001 10
98.66	987	740	0.001 18	0.001 17	0.001 16	0.001 15	0.001 14	0.001 13
101.32	1013	760	0.001 21	0.001 20	0.001 19	0.001 18	0.001 17	0.001 17
103.99	1040	780	0.001 24	0.001 23	0.001 22	0.001 21	0.001 21	0.001 20
106.66	1067	800	0.001 27	0.001 26	0.001 25	0.001 24	0.001 24	0.001 23

Computed for air at 50 % relative humidity using Option A in SOP 2. Conversions of barometric pressure are not exact. See NIST Special Publication 811 for exact conversions.

*Equations to compute the density of air at any pressure, temperature, and relative humidity are given in Section 8 of the Appendix to SOP No. 2 and Option B is preferred for all precision mass and volume calibrations.

Table 9.10 Density and Coefficient of Expansion

Material	Density (g/cm^3)	Coefficient of Expansion		
		Linear (/°C)	Cubical (/°C)	Cubical (/°F)
Aluminum	2.7	0.000 024	0.000 069	0.000 038 *(not suitable for provers)*
Brass	8.4 at 0 °C 8.3909 at 20 °C	0.000 019	0.000 054	
Cast iron	7.0 7.2	0.000 010	0.000 030	
Copper		0.000 017	0.000 050	
Diamond		0.000 001 18	0.000 003 5	
Fused silica (quartz)		0.000 000 5	0.000 001 6	
Glass, borosilicate (T1CA)			0.000 010	
Glass, borosilicate (T1CB)			0.000 015	
Glass, soda-lime			0.000 025	
Gold	18.0		0.000 043	
Invar		0.000 000 4	0.000 001 2	
Lead	11.34	0.000 029	0.000 087	
Length bench		0.000 010 6		
Mercury			0.000 018	
Nichrome	8.39		0.000 039	
Nichrome V	8.5		0.000 039	
Platinum		0.000 011	0.000 027	
Polycarbonate plastic			0.000 45	
Polypropylene plastic			0.000 24	
Polystyrene plastic			0.000 21	
Steel, stainless (mass)	7.84, 7.95, 8.0		0.000 045	
Steel, stainless (provers)		0.000 018	0.000 047 7	0.000 026 5
Steel, tape, mild		0.000 0116	0.000 033 5	
Steel, pressure vessel, low carbon				0.000 016
Steel, prover, low carbon		0.000 012	0.000 033 5	0.000 018 6
Steel, terne plate		0.000 012	0.000 035	0.000 019 5
Tantalum	16.6		0.000 020	
Water (20 °C)			0.000 21	

Reference values for cubical coefficient of expansion given in per °F are provided as reference for prover calibrations used for petroleum products where the reference temperature is 60 °F.

Table 9.11 A Short Table of Random Numbers

46	96	85	77	27	92	86	26	45	21	89	91	71	42	64	64	58	22	75	81	74	91	48	46	18
44	19	15	32	63	55	87	77	33	29	45	00	31	34	84	05	72	90	44	27	78	22	07	62	17
34	39	80	62	24	33	81	67	28	11	34	79	26	35	34	23	09	94	00	80	55	31	63	27	91
74	97	80	30	65	07	71	30	01	84	47	45	89	70	74	13	04	90	51	27	61	34	63	87	44
22	14	61	60	86	38	33	71	13	33	72	08	16	13	50	56	48	51	29	48	30	93	45	66	29
40	03	96	40	03	47	24	60	09	21	21	18	00	05	86	52	85	40	73	73	57	68	36	33	91
52	33	76	44	56	15	47	75	78	73	78	19	87	06	98	47	48	02	62	03	42	05	32	55	02
37	59	20	40	93	17	82	24	19	90	80	87	32	74	59	84	24	49	79	17	23	75	83	42	00
11	02	55	47	48	84	74	36	22	67	19	20	15	92	53	37	13	75	54	89	56	73	23	39	07
10	33	79	26	34	54	71	33	89	74	68	48	23	17	49	18	81	05	52	85	70	05	73	11	17
67	59	28	25	47	89	11	65	65	20	42	23	96	41	64	20	30	89	87	64	37	93	36	96	35
93	50	75	20	09	18	54	34	68	02	54	87	23	05	43	36	98	29	97	93	87	08	30	92	98
24	43	23	72	80	64	34	27	23	46	15	36	10	63	21	59	69	76	02	62	31	62	47	60	34
39	91	63	18	38	27	10	78	88	84	42	32	00	97	92	00	04	94	50	05	75	82	70	80	35
74	62	19	67	54	18	28	92	33	69	98	96	74	35	72	11	68	25	08	95	31	79	11	79	54
91	03	35	60	81	16	61	97	25	14	78	21	22	05	25	47	26	37	80	39	19	06	41	02	00
42	57	66	76	72	91	03	63	48	46	44	01	33	53	62	28	80	59	55	05	02	16	13	17	54
06	36	63	06	15	03	72	38	01	58	25	37	66	48	56	19	56	41	29	28	76	49	74	39	50
92	70	96	70	89	80	87	14	25	49	25	94	62	78	26	15	41	39	48	75	64	69	61	06	38
91	08	88	53	52	13	04	82	23	00	26	36	47	44	04	08	84	80	07	44	76	51	52	41	59
68	85	97	74	47	53	90	05	90	84	87	48	25	01	11	05	45	11	43	15	60	40	31	84	59
59	54	13	09	13	80	42	29	63	03	24	64	12	43	28	10	01	65	62	07	79	83	05	59	61
39	18	32	69	33	46	58	19	34	03	59	28	97	31	02	65	47	47	70	39	74	17	30	22	65
67	43	31	09	12	60	19	57	63	78	11	80	10	97	15	70	04	89	81	78	54	84	87	83	42
61	75	37	19	56	90	75	39	03	56	49	92	72	95	27	52	87	47	12	52	54	62	43	23	13
78	10	91	11	00	63	19	63	74	58	69	03	51	38	60	36	53	56	77	06	69	03	89	91	24
93	23	71	58	09	78	08	03	07	71	79	32	25	19	61	04	40	33	12	06	78	91	97	88	95
37	55	48	82	63	89	92	59	14	72	19	17	22	51	90	20	03	64	96	60	48	01	95	44	84
62	13	11	71	17	23	29	25	13	85	33	35	07	69	25	68	57	92	57	11	84	44	01	33	66
29	89	97	47	03	13	20	86	22	45	59	98	64	53	89	64	94	81	55	87	73	81	58	46	42
16	94	85	82	89	07	17	30	29	89	89	80	98	36	25	36	53	02	49	14	34	03	52	09	20
04	93	10	59	75	12	98	84	60	93	68	16	87	60	11	50	46	56	58	45	88	72	50	46	11
95	71	43	68	97	18	85	17	13	08	00	50	77	50	46	92	45	26	97	21	48	22	23	08	32
86	05	39	14	35	48	68	18	36	57	09	62	40	28	87	08	74	79	91	08	27	12	43	32	03
59	30	60	10	41	31	00	69	63	77	01	89	94	60	19	02	70	88	72	33	38	88	20	60	86
05	45	35	40	54	03	98	96	76	27	77	84	80	08	64	60	44	34	54	24	85	20	85	77	32
71	85	17	74	66	27	85	19	55	56	51	36	48	92	32	44	40	47	10	38	22	52	42	29	96
80	20	32	80	98	00	40	92	57	51	52	83	14	55	31	99	73	23	40	07	64	54	44	99	21
13	50	78	02	73	39	66	82	01	28	67	51	75	66	33	97	47	58	42	44	88	09	28	58	06
67	92	65	41	45	36	77	96	46	21	14	39	56	36	70	15	74	43	62	69	82	30	77	28	77
72	56	73	44	26	04	62	81	15	35	79	26	99	57	28	22	25	94	80	62	95	48	98	23	86
28	86	85	64	94	11	58	78	45	36	34	45	91	38	51	10	68	36	87	81	16	77	30	19	36
69	57	40	80	44	94	60	82	94	93	98	01	48	50	57	69	60	77	69	60	74	22	05	77	17
71	20	03	30	79	25	74	17	78	34	54	45	04	77	42	59	75	78	64	99	37	03	18	03	36
89	98	55	98	22	45	12	49	82	71	57	33	28	69	50	59	15	09	25	79	39	42	84	18	70
58	74	82	81	14	02	01	05	77	94	65	57	70	39	42	48	56	84	31	59	18	70	41	74	60
50	54	73	81	91	07	81	26	25	45	49	61	22	88	41	20	00	15	59	93	51	60	65	65	63
49	33	72	90	10	20	65	28	44	63	95	86	75	78	69	24	41	65	86	10	34	10	32	00	93
11	85	01	43	65	02	85	69	56	88	34	29	64	35	48	15	70	11	77	83	01	34	82	91	04
34	22	46	41	84	74	27	02	57	77	47	93	72	02	95	63	75	74	69	69	61	34	31	92	13

From: NIST Handbook 91, p T-82

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