

SOP 5

**Recommended Standard Operating Procedure
for
Using a 3-1 Weighing Design**

1. Introduction

1.1. Purpose

The 3-1 weighing design is a combination of three double substitution comparisons of three weights of equal nominal value; a standard, an unknown weight, and a second standard called a check standard. (The check standard may be made up of a summation of weights.) The weights are compared using an equal-arm, single-pan mechanical, full electronic, or a combination balance utilizing built-in weights and a digital indication. The specific SOP for the double substitution procedure for each balance is to be followed. The 3-1 weighing design provides two methods of checking the validity of the measurement using an integrated F-test to monitor the within process standard deviation and a t-test to evaluate the stability of the standard and check standard. Hence, the procedure is especially useful for high accuracy calibrations for weights in OIML¹ Classes E₂ to F₂ and ASTM² Classes 0 and 1, in which it is critical to assure that the measurements are valid and well documented. This procedure is recommended as a minimum for precision calibration of laboratory working standards that are subsequently used for lower level calibrations and for routine calibration of precision mass standards used for balance calibration. For surveillance of reference standards, such as OIML E₁, ASTM 00 or better, working standards, and for calibration of precision mass standards used to calibrate other mass standards, see SOP 28 for the use of higher level weighing designs.

1.2. Prerequisites

- 1.2.1. Verify that valid calibration certificates with appropriate values and uncertainties are 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 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 OIML is the International Organization for Legal Metrology. Weight classes are published in OIML R111, 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.

- 1.2.3. The balance that is used must be in good operating condition with sufficiently small process standard deviation as verified by F-test values, pooled short term standard deviations, and by a valid control chart for check standards or preliminary experiments to ascertain its performance quality when new balances are put into service.
- 1.2.4. The operator must be experienced in precision weighing techniques. The operator must have specific training in SOP 2, SOP 4, SOP 5, SOP 29, and be familiar with the concepts in GMP 10.
- 1.2.5. Verify that the laboratory facilities 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 (%)
I	OIML E ₁ , ASTM 000, 00, 0 Lower and upper limits: 18 °C to 23 °C Maximum changes: ± 0.5 °C / 12 h and ± 0.3 °C / h	40 to 60 ± 5 / 4 h
	OIML E ₂ , ASTM 1 Lower and upper limits: 18 °C to 23 °C Maximum changes: ± 1 °C / 12 h and ± 0.7 °C / h	
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

1.2.5.1. It is important that the difference in temperature between the weights and the air inside the mass comparator is 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. 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

³ Echelon I corresponds to weights of Classes OIML E₁ and E₂, Echelon II corresponds to weights of Classes OIML F₁ and F₂.

This method can be performed on any type of balance using the appropriate double substitution SOP for the particular balance. Because considerable effort is involved, this weighing design is most useful for calibrations of the highest accuracy. The weighing design uses three double substitutions to calibrate a single unknown weight. This introduces redundancy into the measurement process and permits two checks on the validity of the measurement; one on accuracy and stability of the standard and the other on process repeatability. A least-squares best fit analysis is done on the measurement outputs to assign a value to the unknown weight. The standard deviation of the process depends upon the resolution of the balance and the care exercised to make the required weighings. The accuracy will depend upon the accuracy and uncertainty of the calibration of the standard weights and the precision of the comparison.

2.2. Summary

A standard weight, S , an unknown weight, X , and a check standard, S_c , are intercompared in a specific order using several double substitution processes. The balance and the weights must be prepared according to the appropriate double substitution SOP for the particular balance being used. Once the balance and weights have been prepared, all readings must be taken from the reading scale of the balance without adjusting the balance or weights in any way. Within a double substitution all weighings are made at regularly spaced time intervals to minimize effects due to instrument drift. The 3-1 weighing design includes air buoyancy corrections.

2.3. Apparatus/Equipment Required

- 2.3.1. Precision analytical balance or mass comparator with sufficient capacity and resolution for the planned calibrations.
- 2.3.2. Calibrated reference standards or 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 to 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.⁴
- 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.⁴

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.

⁴The 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.

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.

2.5. Procedure

2.5.1. Preliminary Procedure

2.5.1.1. Weights are visually inspected for cleanliness and damage. Follow the laboratory policy and customer contract review process 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 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 by blowing air across the surface 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:

Table 3. Cleaning Stabilization.

Weight class	E ₁	E ₂	F ₁	F ₂ to M ₃
After cleaning with alcohol	7 to 10 days	3 to 6 days	1 to 2 days	1 hour
After cleaning with distilled water	4 to 6 days	2 to 3 days	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 F₁ (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. Weights must be completely dry prior to calibration.

Table 4. Minimum Equilibration Times⁵.

ΔT^*	Nominal Mass ⁶	OIML Class E ₁ (time in h)	OIML Class E ₂ (time in h)	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	-	70	33	4
	10, 20, 50 kg	45	27	12	3
	1, 2, 5 kg	18	12	6	2
	100, 200, 500 g	8	5	3	1
	10, 20, 50 g	2	2	1	1
	< 10 g	1	1	1	0.5
± 5 °C	1 000, 2 000, 5 000 kg	-	-	1	1
	100, 200, 500 kg	-	40	4	1
	10, 20, 50 kg	36	18	4	1
	1, 2, 5 kg	15	8	3	1
	100, 200, 500 g	6	4	2	0.5
	10, 20, 50 g	2	1	1	0.5
	< 10 g	0.5	0.5	0.5	0.5
± 2 °C	100 kg to 5 000 kg	-	16	1	0.5
	10, 20, 50 kg	27	10	1	0.5
	1, 2, 5 kg	12	5	1	0.5
	100, 200, 500 g	5	3	1	0.5
	< 100 g	2	1	1	0.5
± 0.5 °C	100 kg to 5 000 kg	-	1	0.5	0.5
	10, 20, 50 kg	11	1	0.5	0.5
	1, 2, 5, kg	7	1	0.5	0.5
	100, 200, 500 g	3	1	0.5	0.5
	< 100 g	1	0.5	0.5	0.5

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

⁵ Consider equivalent ASTM Classes for equilibration times.

⁶ Nominal masses in the 1, 2, 5 combinations include intermediate values such as 3.

2.5.2. Weighing Design Matrix

The following table shows the intercomparisons to be made in the 3-1 design, in a matrix format as shown in NBS Technical Note 952, Designs for the Calibration of Standards of Mass, J. M. Cameron, M. C. Croarkin, and R. C. Raybold, 1977:

Table 5. Weighing Design Matrix.

Weight ID Comparison	S	X	S _c
a ₁	+	-	
a ₂	+		-
a ₃		+	-
Standard	+		
Check Standard			+

This design is represented as design ID “A.1.1” in Technical Note 952, with the exception that the design order is reversed and Restraint B is used⁷. The restraint is another name for the “standard” used in the comparison that may be found in NBS Technical Note 952. This matrix may be useful for data reduction purposes. When creating a data file for this design, the design matrix will appear as the numbers in the following table:

Restraint	1	0	0
Check	0	0	1
Following series sum	0	0	0
Report	0	1	1
1st double sub	1	-1	0
2nd double sub	1	0	-1
3rd double sub	0	1	-1

2.5.3. Measurement Procedure

Record the pertinent information for the standard, S, unknown, X, and check standard, S_c, as indicated on a suitable data sheet such as the one in the Appendix of this SOP. Record the laboratory ambient temperature,

⁷ Additional 3-1 weighing designs are published. This procedure provides the calculations and solutions only for this particular design positions of mass artifacts.

barometric pressure, and relative humidity, before and after each measurement. If performing buoyancy corrections on each measurement, record environmental data before observations 1, 5, 9 and after measurement 12. Perform the measurements in the order shown in the following table.

Table 6. Measurement Procedure.

Double Substitution	Measurement Number	Weights on Pan	Observation
$a_1: S \text{ vs } X$	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
$a_2: S \text{ vs } S_c$	5	$S + t_s$	O_1
	6	$S_c + t_{sc}$	O_2
	7	$S_c + t_{sc} + sw$	O_3
	8	$S + t_s + sw$	O_4
$a_3: X \text{ vs } S_c$	9	$X + t_x$	O_1
	10	$S_c + t_{sc}$	O_2
	11	$S_c + t_{sc} + sw$	O_3
	12	$X + t_x + sw$	O_4

3. Calculations

- 3.1. Calculate the average air density, ρ_A , as described in the Appendix to NISTIR 6969, SOP No. 2, Option B.
- 3.2. Calculate the measured differences, a_1 , a_2 , and a_3 , for the weights used in each double substitution using the following formula (note: do not confuse this formula with the calculations used in SOP 4, NISTIR 6969; the signs will be opposite SOP 4, Option A.):

$$a_x = \frac{(O_1 - O_2 + O_4 - O_3)}{2} \frac{M_{sw} \left(1 - \frac{\rho_A}{\rho_{sw}} \right)}{O_3 - O_2}$$

- 3.3. Calculate the within process standard deviation, s_w , for the 3-1 weighing design. This

standard deviation has one degree of freedom.

$$s_w = 0.577(a_1 - a_2 + a_3)$$

- 3.4. Calculate the F statistic which compares the within process standard deviation, s_w , to the pooled within process standard deviation (or accepted within process standard deviation). (See chapter 8.4 and 8.5 in NISTIR 6969, for a discussion of the statistics used in weighing designs.)

$$F - \text{statistic} = \frac{s_w^2}{(\text{Pooled } s_w)^2}$$

The F-statistic so computed must be less than the F-value obtained from an F-table at 99 % confidence level (Table 9.5, NISTIR 6969) to be acceptable. The F-value is obtained from the F-table for numerator degrees of freedom equal one, and denominator degrees of freedom equal to the number of degrees of freedom in the pooled within process standard deviation. If the data fails the F-test and the source of the error cannot be determined conclusively, the measurement must be repeated.

- 3.5. Calculate the observed mass value of the check standard.

Calculate the least-squares measured difference d_{sc} for S_c .

$$d_{sc} = \frac{-a_1 - 2a_2 - a_3}{3}$$

- 3.6. Calculate the observed mass of S_c , M_{sc} .

$$M_{sc} = \frac{M_s \left(1 - \frac{\rho_A}{\rho_s} \right) + d_{sc} + M_{ts} \left(1 - \frac{\rho_A}{\rho_{ts}} \right) - M_{tsc} \left(1 - \frac{\rho_A}{\rho_{tsc}} \right)}{\left(1 - \frac{\rho_A}{\rho_{sc}} \right)}$$

- 3.6.1. Calculate the Conventional Mass of S_c , CM_{Sc} :

$$CM_{Sc} = \frac{M_{Sc} \left(1 - \frac{0.0012}{\rho_{Sc}} \right)}{0.999850}$$

- 3.7. Evaluate the mass M_{sc} , or conventional mass CM_{sc} , of S_c .

The mass or conventional mass is evaluated with a t-test in the procedure based on comparison with the accepted mass or conventional mass value. The mass or conventional mass (depending on what is tracked in the laboratory) is also 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.

Calculate E_n to compare the mean mass value of the check standard, M_{sc} from the 3-1 design to the accepted and calibrated reference value (R_{sc}) that has adequate metrological traceability for the check standard.

$$E_n = \frac{|\overline{M}_{sc} - R_{sc}|}{\sqrt{U_{M_{sc}}^2 + U_{R_{sc}}^2}}$$

The E_n value must be less than one to pass.

- 3.8. Calculate the least-squares measured difference, d_x , for X .

$$d_x = \frac{-2a_1 - a_2 + a_3}{3}$$

- 3.9. Calculate the mass of X , M_x .

$$M_x = \frac{M_s \left(1 - \frac{\rho_A}{\rho_s} \right) + d_x + M_{ts} \left(1 - \frac{\rho_A}{\rho_{ts}} \right) - M_{tx} \left(1 - \frac{\rho_A}{\rho_{tx}} \right)}{\left(1 - \frac{\rho_A}{\rho_x} \right)}$$

- 3.10. Calculate the conventional mass⁸ of X , CM_x . The conventional mass should be reported.

3.10.1. Conventional mass

⁸ 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/cm³; reference temperature 20 °C; *normal* air density 0.0012 g/cm³. Conventional mass was formerly called “Apparent Mass versus 8.0 g/cm³” in the United States. See *OIML D28 (2004)*.

$$CM_x = \frac{M_x \left(1 - \frac{0.0012}{\rho_x} \right)}{0.999850}$$

3.10.2. 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.) Apparent mass versus brass (8.3909 g/cm³ at 20 °C)

$$AM_x \text{ vs. } 8.4 = \frac{M_x \left(1 - \frac{0.0012}{\rho_x} \right)}{\left(1 - \frac{0.0012}{8.3909} \right)}$$

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 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. 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, "Standard Operating Procedures for the Assignment of Uncertainty" (NISTIR 6969), for the complete standard operating

procedure for calculating the uncertainty.

When the 3-1 weighing design is used in conjunction with specialized software for data reduction, see SOP 28, “Recommended Standard Operating Procedure for Using Advanced Weighing Designs”, for detailed instructions on calculating the uncertainty components which are required.

- 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. Usually only one standard is used as the restraint for the 3-1 weighing design, the uncertainty of the check standard is not included in assigning an uncertainty to the unknown mass.
- 5.2. The value for the standard deviation of the measurement process s_p is obtained from the control chart data for check standards using only 3-1 weighing design measurements (see SOP No. 9.) The within-process standard deviation, s_w , is only used as a part of the process variability evaluation using the F-test unless between time components are also determined. In that case, the standard deviation of the process, s_p , is treated as s_t (standard deviation over time) and see NISTIR 5672, SOP 28 for details. Statistical control must be verified by the measurement of the check standard in the 3-1 design.
 - 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 should be used.
 - 5.2.2. For SOP 5 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:

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

Because SOP 5 uses an average of the observations from redundant weighing designs, a smaller value may be used when the standard deviation of the process is less than the balance resolution. 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, 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. Note: this may be done using a simplified “baseline approach” or a Kragten analysis.⁹
- 5.3.3. Option 3. In the event that buoyancy corrections are not performed, include an uncorrected systematic standard uncertainty due to the 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

⁹ 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.

Uncertainty Component Description	Symbol	Source	Typical Distribution
Air temperature (for air density)	u_t	SOP 2 or OIML R111	Rectangular
Air pressure (for air density)	u_p	SOP 2 or OIML R111	Rectangular
Air relative humidity (for air density)	u_{RH}	SOP 2 or OIML R111	Rectangular
Air density (formula)	u_{ρ_a}	SOP 2 or OIML R111	Rectangular
Mass densities (5.4)	u_{ρ_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 E 617 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 (e.g.,)

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 3-1 Weighing Design When Tare Weights Are Used

Laboratory data and conditions:

Operator			Before	After
Date		Temperature °C		
Balance		Barometric Pressure mm Hg		
Load		Relative Humidity %		
Pooled within process s.d., $s_w =$		Degrees of freedom for process s.d.		
Check standard s.d., $s_p =$		Degrees of freedom from control chart		

Mass standard(s) data:

ID (Insert Set or SN)	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					

Laboratory observations:

Balance Observations, Units _____					
Time:					
$S - X = a_1$		$S - S_c = a_2$		$X - S_c = a_3$	
$S + t_s$		$S + t_s$		$X + t_x$	
$X + t_x$		$S_c + t_{sc}$		$S_c + t_{sc}$	
$X + t_x + sw$		$S_c + t_{sc} + sw$		$S_c + t_{sc} + sw$	
$S + t_s + sw$		$S + t_s + sw$		$X + t_x + sw$	
Time:					
$a_1 =$		$a_2 =$		$a_3 =$	

Appendix A – Flow Chart of the 3-1 Weighing Design Process

