

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 reference 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. (These procedures are published in NISTIR 6969, Selected Laboratory and Measurement Practices, and Procedures to Support Basic Mass Calibrations.)
- 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.

Echelon³	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

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.

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

2 Methodology

2.1 Scope, Precision, Accuracy

This method can be performed on any type of balance or mass comparator using the appropriate double substitution method for the weighing instrument. 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 method for the 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 during the process. 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 NISTIR 6969, 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.

- 2.3.6 Forceps and gloves must be selected to avoid damage or contamination to mass standards.
- 2.3.7 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.
- 2.3.8 Calibrated barometer with sufficiently small resolution, stability, and uncertainty (See NISTIR 6969, SOP 2, e.g., accurate to ± 66.5 Pa (0.5 mmHg)) to determine barometric pressure.⁴
- 2.3.9 Calibrated thermometer with sufficiently small resolution, stability, and uncertainty (see SOP 2, e.g., accurate to ± 0.10 °C) to determine air temperature.⁴
- 2.3.10 Calibrated hygrometer with sufficiently small resolution, stability, and uncertainty (see SOP 2, e.g., accurate to ± 10 percent) to determine relative humidity.⁴

⁴NISTIR 6969 includes SOP 2 for the calculation of air density. 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.

2.4 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

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 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 time.

Weight class	E₁	E₂	F₁	F₂ to M₃
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	1 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. Specifically, 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 times.⁵

ΔT^a	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	-	-	24	1
	100, 200, 500 kg	-	40	10	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

^a ΔT = Initial difference between weight temperature and laboratory temperature.

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:

⁵ Consider equivalent ASTM Classes for equilibration times.

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

Table 5. Weighing Design Matrix.

Weight ID Comparison	S	X	S _c
<i>a</i> ₁	+	-	
<i>a</i> ₂	+		-
<i>a</i> ₃		+	-
Standard (Restraint)	+		
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. The following table may be useful for data reduction purposes if using the NIST Mass Code. When creating a data file for this design, the restraint, check, and design vectors will appear in the order and as the numbers in the following table:

Table 6. Weighing Design Vector for Mass Code.

Row in Mass Code Design	First Entry	Second Entry	Third Entry
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

Perform the measurement as a series of 3 double substitutions. Review SOP 4 (NISTIR 6969) if needed. 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. Measure the laboratory ambient temperature, barometric pressure, and relative humidity, before and after the set of three double substitutions (i.e., before and after all twelve observations). If performing buoyancy corrections on *each* double substitution within the design, record environmental data before observations 1, 5, 9 and after measurement 12. Perform the mass measurements in the order shown in the following table.

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

Table 7. 12 Measurement Observations.

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

Note: As the NIST Mass Code software can be used to perform the calculations for this process, the balance of this section is provided as reference to be used for manual or computerized calculations using other software packages.

- 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.):

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

- 3.3 Calculate the within process standard deviation, s_w , for the 3-1 weighing design. This standard deviation has one degree of freedom.

$$s_w = 0.577(a_1 - a_2 + a_3) \quad \text{Eqn. (2)}$$

- 3.4 Calculate the F statistic which compares the observed within process standard deviation, s_w , to the pooled (accepted) within process standard deviation. (See NISTIR 6969, Sections 8.4, 8.5, and 8.9.2, for a discussion of the statistics used in weighing designs.)

$$F = \frac{s_w^2 \text{ Observed}}{s_w^2 \text{ Accepted}} \quad \text{Eqn. (3)}$$

The calculated F-statistic must be less than the F-value obtained from an F-table at 95 % confidence level (Table 9.12, 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 least-squares measured difference d_{sc} for S_c .

$$d_{sc} = \frac{-a_1 - 2a_2 - a_3}{3} \quad \text{Eqn. (4)}$$

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

$$M_{sc} = \frac{M_s \left(1 - \frac{\rho_A}{\rho_s} \right) + d_{sc} + M_{ts} \left(1 - \frac{\rho_A}{\rho_{ts}} \right) - M_{tsc} \left(1 - \frac{\rho_A}{\rho_{tsc}} \right)}{\left(1 - \frac{\rho_A}{\rho_{sc}} \right)} \quad \text{Eqn. (5)}$$

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

$$CM_{Sc} = \frac{M_{sc} \left(1 - \frac{0.0012}{\rho_{sc}} \right)}{0.999850} \quad \text{Eqn. (6)}$$

- 3.8 Evaluate the mass M_{Sc} , or conventional mass CM_{Sc} , of S_c .

The observed mass or conventional mass is evaluated with a t-test in the procedure based on comparison with the accepted mass or conventional mass value determined from the mean of the control chart. 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.

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

$$d_x = \frac{-2a_1 - a_2 + a_3}{3} \quad \text{Eqn. (7)}$$

3.10 Calculate the mass of X , M_x .

$$M_x = \frac{M_s \left(1 - \frac{\rho_A}{\rho_s}\right) + d_x + M_{t_s} \left(1 - \frac{\rho_A}{\rho_{t_s}}\right) - M_{t_x} \left(1 - \frac{\rho_A}{\rho_{t_x}}\right)}{\left(1 - \frac{\rho_A}{\rho_x}\right)} \quad \text{Eqn. (8)}$$

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

$$CM_x = \frac{M_x \left(1 - \frac{0.0012}{\rho_x}\right)}{0.999850} \quad \text{Eqn. (9)}$$

3.12 Calculate apparent mass versus brass only if requested. 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)

$$AM_{x \text{ vs. } 8.4} = \frac{M_x \left(1 - \frac{0.0012}{\rho_x}\right)}{\left(1 - \frac{0.0012}{8.3909}\right)} \quad \text{Eqn. (10)}$$

4 Measurement Assurance

4.1 The within process standard deviation is incorporated into the NIST Mass Code and is used to conduct an F-test of the observed standard deviation versus the pooled/accepted standard deviation of the process at a 95 % confidence level. If calculations are performed manually or using other software, follow the process described in section 3 for F-test evaluation.

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)*).

- 4.2 SOP 5 weighing design integrates a suitable check standard (See NISTIR 6969, GLP 1, SOP 9, and SOP 30).
- 4.3 The check standard value is calculated and immediately evaluated on the control chart to verify that the mass is within established limits. A t-test may be incorporated to check the observed value of the check standard against the accepted value. It is evaluated using the following equation and a 95 % confidence level. All values must be entered in the control chart, even if failing this statistic to ensure the variability obtained for the process is truly representative of the process and not unduly reduced over time. The observed value of the check standard is compared to the accepted mean value of the check standard and divided by the standard deviation for the check standard observations over time. This equation monitors stability over time but should not be used to assess for bias. A calculated t-value less than two is within the warning limits of the process. A calculated t-value between two and three represents a value between the warning limits and control/action limits. A calculated t-value exceeding three represents a value outside of the control/action limits and suitable action must be taken. Calculated values of the t-statistic may also be monitored over time to determine the presence of drift.

$$t = \frac{(S_c - \bar{S}_c)}{s_p} \quad \text{Eqn. (11)}$$

- 4.4 Check standard measurement results obtained over time are used to calculate the standard deviation of the measurement process, s_p .
- 4.5 The mean value of the check standard over time is also compared to an appropriate reference value of the check standard with respect to applicable expanded uncertainties to evaluate bias and drift over time. Excessive drift or bias must be investigated and followed with suitable corrective action. (See NISTIR 6969, SOP 9, Section 4.2 for assessment methodology.)

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 NISTIR 6969, SOP 29, “Standard Operating Procedures for the Assignment of Uncertainty”, 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. Where the coverage factor or confidence interval is not given, the laboratory should either contact the calibration provider to obtain the correct divisor or use a value of $k = 2$, assuming that the expanded uncertainty was reported with an approximate 95 % confidence interval (95.45 %).
- 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 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 from the control chart is less than the resolution of the balance being used, the laboratory may round up to the nearest balance division to represent the standard deviation, or use the larger of the standard deviation of the process and one of the following estimates for repeatability is used to represent the standard deviation of the process:
- 5.2.1.1 If the laboratory prefers a conservative approach, or when the current and representative degrees of freedom are less than 30, the larger of the control chart s_p and the result from Eqn. 12 should be used, where d is the smallest balance division. For example, if the balance division is 0.1 mg, the smallest standard deviation may be 0.06 mg. If the laboratory calculated standard deviation is 0.075 mg, then 0.075 mg is used.

$$s_p = \frac{d}{\sqrt{3}} \approx 0.6d \quad \text{Eqn. (12)}$$

5.2.1.2 When the laboratory has the confidence associated with a well characterized measurement process and has 30 or more degrees of freedom to represent the process, the larger of the observed s_p or Eqn. 13 is used. For example, if the balance division is 0.1 mg, the smallest standard deviation may be 0.03 mg. If the laboratory observed standard deviation is larger than 0.03 mg, then that is the value to be used.

$$s_p = \frac{d}{2\sqrt{3}} \approx 0.3d \quad \text{Eqn. (13)}$$

- 5.3 Uncertainty due to air buoyancy corrections and air density. Select one of the following options in priority preference for calculating the uncertainty associated with air buoyancy.
- 5.3.1 Option 1, preferred. 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 quantifying 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 “what if” approach or a Kragten analysis.⁹
- 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 may be included if corrective action is not taken. (See NISTIR 6969, SOP 29 for additional details.)
- 5.6 Example components to be considered for an uncertainty budget table are shown in the following table.

⁹ A baseline “what if” 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).

Table 8. Example Uncertainty Budget Table.

Uncertainty Component Description	Symbol	Source	Typical Distribution
Uncertainty of the standard mass(es) (5.1)	u_s	Calibration certificate	Expanded U divided by coverage factor
Accepted standard deviation of the process (5.2, 5.2.1)	s_p	Control chart, standard deviation chart OR estimates when s_p is smaller than balance division	Normal OR Rectangular, one-half rectangular
Uncertainty of the air buoyancy correction (5.3)	u_b	OIML R111	Rectangular
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	NISTIR 6969 SOP 29

5.7 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 the uncertainty associated with the 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).

NOTE: Where inadequate degrees of freedom are available, k , is determined using the appropriate degrees of freedom and the 95.45 % column in the table from Appendix A of NISTIR 6969, SOP 29.

6 Certificate

6.1 Report results as described in NISTIR 6969, SOP No. 1, Preparation of Calibration Certificates. Report the mass, conventional mass, environmental conditions during the calibrations, mass density used (reported, measured, or assumed (specify which applies)), and calculated expanded uncertainties with coverage factor(s).

6.2 Conformity assessments.

Evaluate compliance to applicable tolerances as needed or required by the customer or by legal metrology requirements. Decision criteria for uncertainty and tolerance evaluations include two components: 1) the expanded uncertainty, U , must be $< 1/3$ of the applicable tolerances published in ASTM E 617 and OIML R111 documentary standards and 2) the absolute value of the conventional mass correction value plus the expanded uncertainty must be less than the applicable tolerance to confidently state that mass standards are in or out of tolerance. Compliance assessments must note the applicable documentary standard and which portions of the standard were or were not evaluated.

Appendix A
Observation Sheet for
3-1 Weighing Design When Tare Weights Are Used

Laboratory data and conditions:

Operator		Before	After
Date		Temperature °C	
Balance		Barometric Pressure mmHg	
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	

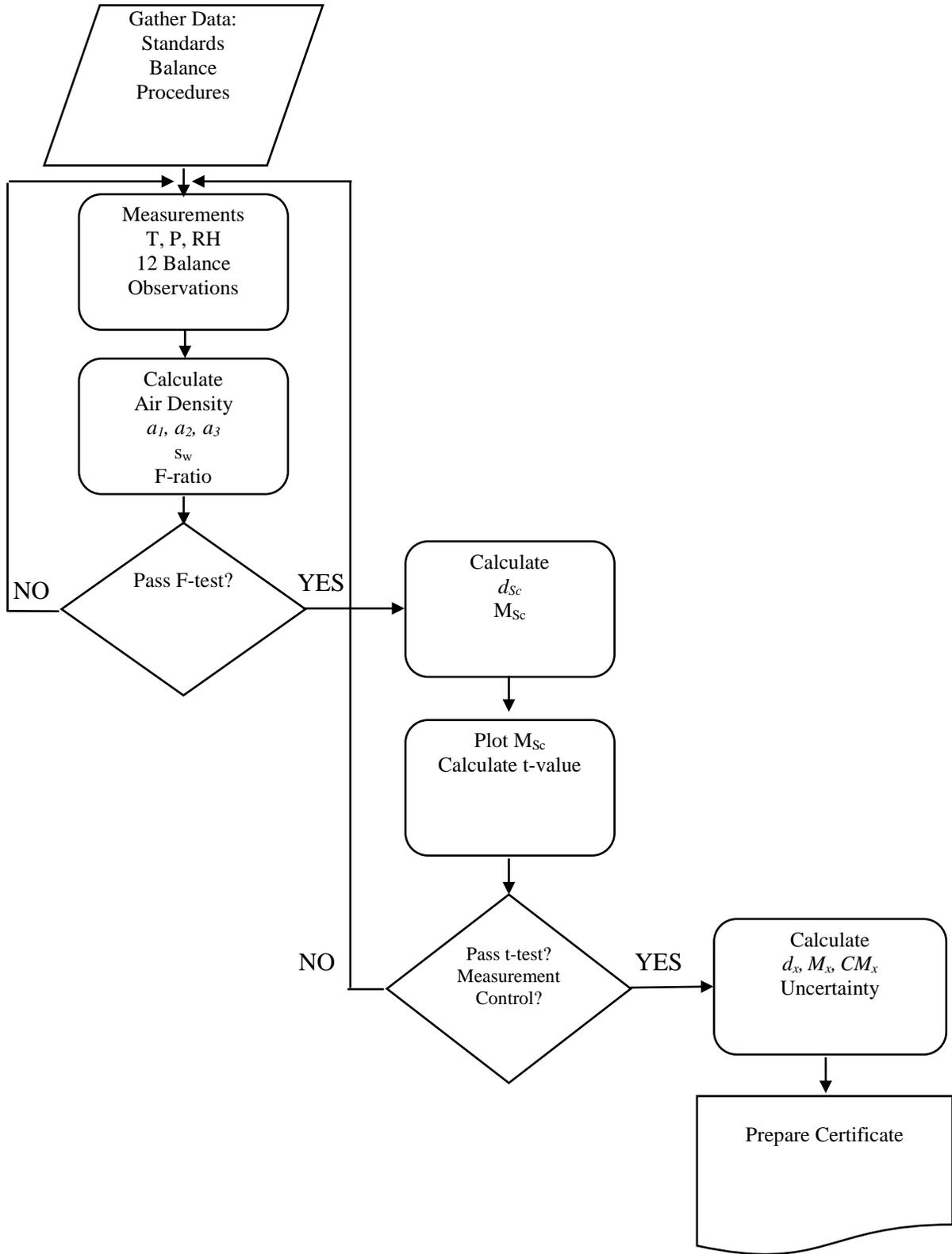
Mass standard(s) data:

ID (Insert Set or SN)	Nominal	Mass Correction	Expanded Unc: from cal. certificate	Unc: k factor	Density g/cm^3	Unc Density $k=1$
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 B – Flow Chart of the 3-1 Weighing Design Process



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Figure 1. Appendix B – Flow Chart of the 3-1 Weighing Design Process.