

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 (OIML¹ Classes F₁ to M₃, ASTM² 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 F₁ and F₂. Echelon III corresponds to weights of Classes OIML M₁, M₂, and M₃. This procedure does not provide adequate redundancy for OIML Classes E₁ or E₂ 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

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.

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 times⁵.

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.

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 mass⁶ 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/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)*.

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

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

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

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:

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_{S_c}					
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)	$S_c + t_{S_c}$	
3 (O_3)	$S_c + t_{S_c} + sw$	
Time:		
Check Standard in Control?	____ Yes ____ No	

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

Laboratory data and conditions:

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_{S_c}					
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)	$S_c + t_{S_c}$	
3 (O_3)	$S_c + t_{S_c} + sw$	
Time:		
Check Standard in Control?	____ Yes ____ No	