SOP No. 7

Recommended Standard Operations 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

1.0   Introduction

   1.1.   Purpose

   In the single substitution procedure a standard and an unknown weight are
   intercompared once to determine the difference in weights. Errors in any built-in
   weights or in the balance indications are eliminated 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 is required and as a single substitution, 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;
   additional precautions must be taken.

   1.2.   Prerequisites

   1.2.1.   Verify that valid calibration certificates are available for the standards
            used in the test.

   1.2.2.   Verify that the standards to be used have sufficiently small standard
            uncertainties for the intended level of calibration. Primary standards
            should not be used at this level.

   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.

   1.2.4.   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.   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	Relative Humidity (%)
II	20 °C to 23 °C, a set point ± 2 °C, maximum change 1.0 °C/h	40 to 60 ± 10 / 4 h
III	18 °C to 27 °C, maximum change 2.0 °C/h	40 to 60 ± 20 / 4 h

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

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 balance with sufficient capacity and sensitivity for the calibrations planned.

2.3.2. Calibrated working standard and sensitivity weights with recent calibration values that are traceable to NIST.

2.3.3. Calibrated small standard weights with recent calibration values that are traceable to NIST to be used as tare weights.

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

2.3.5. Forceps to handle the weights, or gloves to be worn if the weights are moved by hand.

2.3.6. Stop watch or other timing device to observe the time of each measurement.

2.3.7. Calibrated barometer accurate to ± 66.5 Pa (0.5 mm Hg) with recent calibration values that are traceable to NIST to determine air pressure.

2.3.8. Calibrated thermometer accurate to ± 0.10° C with recent calibration values that are traceable to NIST to determine air temperature.

2.3.9. Calibrated hygrometer accurate to ± 10 % with recent calibration values that are traceable to NIST to determine relative humidity.[1]

2.4. Symbols

Table 2.   Symbols used in this procedure

Symbol	Description
S	standard weight
X	weight calibrated
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

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[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.

2.5.   Procedure

2.5.1. Preliminary Procedure

2.5.1.1. Place the test weight and standards in the balance chamber or near the balance overnight to permit the weights and the balance to attain thermal equilibrium.

2.5.1.2. Conduct preliminary measurements to obtain an approximate value for the difference between the standard and the unknown, to determine where the readings occur on the balance, to determine if tare weights are required, to determine the sensitivity weight that must be used, and to determine the time interval required for the balance indication to stabilize.

Tare weights are rarely needed for high precision mass standards. If 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 evaluated in the process of determining 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 GMP 14, Table 1). Select t_s and t_x such that the difference in mass between $S + t_s$ and $X + t_x$ is:

a. Single-pan mechanical balance - less than $\dfrac{1}{10}$ the range of the optical scale.

b. Full electronic balance - less than 0.05 % of the balance capacity.

c. Combination balance - less than $\dfrac{1}{10}$ the range of the digital indications.

A sensitivity weight must be used on equal-arm balances, and is normally used on single-pan mechanical and electronic balances, to ensure that the differences determined through the use of the optical scale or electronic range have valid accuracy and traceability. (e.g., The optical scale is *calibrated* each time the procedure is used through the use of a sensitivity weight). The uncertainty of the sensitivity weight does not need to be included in calculations of uncertainty since the uncertainty value is distributed across the range of use.

If a sensitivity weight will be used, select one that is (See GMP 14, Table 2):

    a. Single-pan balance - between ¼ and ½ the range of the optical scale, and at least 4 times the mass difference between *X* and *S*.

    b. Full electronic balance - at least 4 times the mass difference between *X* and *S* but not exceeding 1 % of the balance capacity.

    c. Combination balance - between ¼ and ½ the range of the digital indications, and at least 4 times the mass difference between *X* and *S*.

2.5.1.3. 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.

2.5.1.4. 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.

2.5.1.5. 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 3.   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 $\pm$ 2 sd of the balance. If this difference is exceeded, reject the data and take a new series of measurements that will so agree.

2.5.3. Measurement Procedure, Optional Sequence B (XSS)

Table 4.   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

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. No air buoyancy correction. 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), 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 no tare weights, and equal nominal values are used, those terms may all be deleted from the equations.

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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. 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[2] of *X*, CM_x. It is recommended that the conventional mass be reported.

3.2.4.1.   Conventional mass

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

3.2.5.   If requested, the apparent mass versus the reference density of brass may be calculated. This value should only be used when calibrating mechanical balances that have been adjusted to this reference density.

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[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. *See OIML IR 33 (1973, 1979), under revision.*

3.2.5.1. Apparent mass versus brass

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

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 of the check standard is used 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.

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 report. 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. Other standard uncertainties usually included at this calibration level include uncertainties associated with calculation of air density and standard uncertainties associated with the density of the standards used.

5.4. Evaluation of compliance. The expanded uncertainty, U, must be $\leq$ 1/3 of the tolerance applicable as per ASTM E 617-97 and OIML R 111 if compliance statements are used

6.   Report

Report results as described in SOP No. 1, Preparation of Calibration/Test Reports.

Appendix
Single Substitution Data Sheet
(Optional Sequence A)
SXX

Laboratory data and conditions:

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

Mass standard(s) data:

ID	Nominal	Mass Correction	Unc: From cal. report	Unc: k factor	Density g/cm^3
S					
X					
sw					
t_s					
t_x					

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:		

Note: dotted line represents decimal point.

Appendix
Single Substitution Data Sheet
(Optional Sequence B)
XSS

Laboratory data and conditions:

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

Mass standard(s) data:

ID	Nominal	Mass Correction	Unc: From cal. report	Unc: k factor	Density g/cm^3
X					
S					
sw					
t_s					
t_x					

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:		

Note: dotted line represents decimal point.

Example: Without Buoyancy Corrections
Single Substitution Data Sheet
(Optional Sequence A)
SXX

Laboratory data and conditions:

Operator				HO
Date	8/27/86	Temperature		22.6 °C
Balance	H 20	Pressure		751.7 mm Hg
Load	50 g	Relative Humidity		50 %
Standard deviation of the process, from control chart, s_p				0.018 mg

Mass standard(s) data:

ID	Nominal	Mass Correction	Unc: From cal. report	Unc: k factor	Density g/cm^3
S	50 g	0.255 mg	0.033 mg	3	8.00
X	50 g				7.95
sw	50 mg	-0.084 mg	0.000 85 mg	2	8.5
t_s					
t_x					
S_c	50 g	0.315 mg	0.045 mg	2	8.00

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 _____ mg _____
Time:	3:40 PM	
1 (O_1)	$S + t_s$	12 ┊ 62
2 (O_2)	$X + t_x$	12 ┊ 51
3 (O_3)	$X + t_x + sw$	62 ┊ 37
Time:	3:45 PM	

Measurement Assurance (Duplication of the Process):

Measurement #	Weights	Balance Observations, Units ____ mg ____
Time:	3:50 PM	
1 (O_1)	$S + t_s$	12 ┊ 67
2 (O_2)	$S_c + t_{Sc}$	12 ┊ 73
3 (O_3)	$S_c + t_{Sc} + sw$	62 ┊ 60
Time:	3:55 PM	

Note: dotted line represents decimal point.

Calculation of Conventional Mass

Use equation 3.1.1 for optional sequence A (*SXX*) without buoyancy corrections[3]:

Since no tare weights were used and equal nominal values were used, the equation may be simplifies as follows:

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

$$C_x = 0.255\,mg + (12.51 - 12.62) \left[\frac{49.916\,mg}{(62.37 - 12.51)} \right]$$

$$C_x = 0.255\,mg + (-0.110\ 124)\,mg$$

$$C_x = 0.144\ 876\,mg$$

Calculation of Uncertainty

Calculate the uncertainty for the calibration:

$$U = u_c * 2$$

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

The uncertainty for the standard from the calibration report (or data sheet) must be divided by the k factor to determine the u_s. Refer to SOP 29 for the use of multiple standards. An additional uncertainty for not performing the air buoyancy correction can be determined using the magnitude of the air buoyancy correction from SOP 2.

$$u_c = \sqrt{ (0.011)^2 + (0.018)^2 + (0.0010)^2 }$$

$$u_c = 0.021\ 330\ 7\,mg$$

$$U = 0.021\ 330\ 7 * 2 = 0.042\ 661\ 5\,mg$$

Uncertainty Statement

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 % confidence interval. Factors not considered in the evaluation: magnetism (weights are considered to meet magnetism specifications unless measurement aberrations are noted), balance eccentricity and

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[3]   Keep in mind that these equations may be truncated for the purpose of this example and minor differences may be seen in the ending decimal places due to the use of calculators or spreadsheets.

linearity (these factors are considered as a part of the measurement process when obtaining the standard deviation of the process).

Compliance Evaluation

You may need to evaluate the conventional mass correction and its uncertainty to determine if a weight is or is not within specified tolerances. The magnitude of the expanded uncertainty must be less than 1/3 of the tolerance to be able to perform that evaluation, according to ASTM E 617-97 and OIML R111 documentary standards.

Load = 50 g

ASTM E 617		OIML R111	
Class	Tolerance (mg)	Class	Tolerance (mg)
1	0.12	E_2	0.10
2	0.25	F_1	0.30

If we look at three times the calculated expanded uncertainty: 0.043 mg x 3 = 0.129 mg, we observe that the uncertainty complies with the 1/3 rule for ASTM Class 2, and OIML Class F_1 but not ASTM Class 1 or OIML Class E_2.

Next, the weight value is considered to be within tolerance when the absolute value of its error plus its uncertainty, do not exceed the tolerance established for the particular class of weight.

Value and uncertainty: *0.145* mg ± 0.043 mg.
0.145 mg + 0.043 mg = 0.188 mg (upper limit of the value, or error bar if graphed)
0.145 mg – 0.043 mg = 0.102 mg (lower limit of the value, or error bar if graphed)

We can see that the correction of 0.145 is within these limits: *0.102 $\leq C_x \leq$ 0.188*, therefore, the value also complies with ASTM Class 2 and OIML Class F_1, and a compliance statement may be included on the calibration report.

Reporting

The conventional mass correction and uncertainty are reported as follows:

C_x = *0.145* mg ± 0.043 mg