

**SOP No. 4****Recommended Standard Operating Procedure  
for  
Weighing by Double 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

The double substitution procedure is one in which a standard and an unknown weight of equal nominal value are compared twice to determine the average difference between the two weights. Errors in any built-in weights or in the 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. Accordingly, the procedure is especially useful for high accuracy calibrations. The procedure does not incorporate measurement control steps to ensure the validity of the standards and the measurement process; therefore, additional precautions must be taken.

## 1.2. Prerequisites

- 1.2.1. Verify that valid calibration certificates are available for the working standards used in the test.
- 1.2.2. Verify that the working standards to be used have sufficiently small standard uncertainties for the intended level of calibration. Primary laboratory reference standards should not be used at this level.
- 1.2.3. Verify that the balance is in good operating condition with sufficiently small process standard deviation as verified by a valid control chart or preliminary experiments to ascertain the performance quality when a new balance is put into service.
- 1.2.4. Verify that the operator is experienced in precision weighing techniques and has had specific training in SOP 2, SOP 4, SOP 29, GMP 4, and 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.

**Table 1. Environmental conditions.**

Echelon <sup>1</sup>	Temperature	Relative Humidity (percent)
II	20 °C to 23 °C, a set point $\pm 2$ °C, maximum change 1.0 °C/h	40 to 60 $\pm 10$ / 4 h
III	18 °C to 27 °C, maximum change 2.0 °C/h	40 to 60 $\pm 20$ / 4 h

## 2. Methodology

### 2.1. Scope, Precision, Accuracy

This method is applicable to all weighings utilizing a mass comparator, 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 will depend upon the sensitivity of the balance and the care exercised to make the required weighings. The accuracy achievable with this procedure depends on the accuracy of the calibration of the working standards and the 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 standard and the same sensitivity weight are then weighed. The latter two weighings provide both second weighings of the standard and the test weight as well as a determination of the sensitivity of the balance under the load conditions at the time of the intercomparison. All weighings are made at regularly spaced time intervals to average out any effects due to instrument drift.

The double 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 differs 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.

<sup>1</sup> Echelon II corresponds to weights of Classes OIML F<sub>1</sub> and F<sub>2</sub>. Echelon III corresponds to weights of Classes OIML M<sub>1</sub>, M<sub>2</sub>, and M<sub>3</sub>. This procedure does not provide adequate redundancy for OIML Classes E<sub>1</sub> or E<sub>2</sub> and SOP 5 or SOP 28 should be used.

- 2.3.2. Calibrated working standard and sensitivity weights with recent calibration values and which 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.
- 2.3.3. Calibrated small standard weights with recent calibration certificates which 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 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 (calibration not required; this is used to ensure consistent timing of the measurement).
- 2.3.7. Calibrated barometer accurate to  $\pm 66.5$  Pa (0.5 mm Hg) with recent calibration values which 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 to determine air pressure.
- 2.3.8. Calibrated thermometer accurate to  $\pm 0.10$  °C with recent calibration values which 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 to determine air temperature.
- 2.3.9. Calibrated hygrometer accurate to  $\pm 10$  percent with recent calibration values which 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 to determine relative humidity.<sup>2</sup>

#### 2.4. Symbols

**Table 2. Symbols used in this procedure.**

<b>Symbol</b>	<b>Description</b>
<i>S</i>	standard reference weight
<i>X</i>	weight to be calibrated

<sup>2</sup>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.

$t$	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.
$\rho_a$	density of air at time of calibration
$\rho_n$	density of normal air (1.2 kg/m <sup>3</sup> )
$\rho$	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.

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	F1	F2 to M3
After cleaning with alcohol	1–2 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. 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. Equilibration Times.**

$\Delta T^*$	Nominal Mass	OIML Class F1 (time in h)	OIML Class F2 to M3 (time in h)
$\pm 20\text{ }^\circ\text{C}$	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
$\pm 5\text{ }^\circ\text{C}$	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

$\Delta T^*$	Nominal Mass	OIML Class F1 (time in h)	OIML Class F2 to M3 (time in h)
	100, 200, 500 g	2	1
	< 100 g	1	1
$\pm 2\text{ }^\circ\text{C}$	<100 g to 5 000 kg	1	0.5
$\pm 0.5\text{ }^\circ\text{C}$	<100 g to 5 000 kg	0.5	0.5

\* $\Delta T$  = 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 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 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 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:

- Single-pan mechanical balance - less than  $\frac{1}{10}$  the range of the optical scale.
- Full electronic balance - less than 0.05 percent of the balance capacity.
- Combination balance - less than  $\frac{1}{10}$  the range of the digital indications.
- Mass comparator – less than  $\frac{1}{10}$  digital range

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 measured differences determined through the use of the optical scale or electronic range have valid accuracy and traceability (See GMP 14, Table 2). (e.g., The optical scale is *calibrated* each time the procedure is performed through the use of a sensitivity weight). The uncertainty of the sensitivity weight does not generally need to be included in calculations of uncertainty since the uncertainty value is distributed across its range of use.

If a sensitivity weight will be used, select one that is:

- a. Single-pan balance - between  $\frac{1}{4}$  and  $\frac{1}{2}$  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 percent of the balance capacity.
- c. Combination balance - between  $\frac{1}{4}$  and  $\frac{1}{2}$  the range of the digital indications, and at least 4 times the mass difference between  $X$  and  $S$ .
- d. Mass comparator – at least 4 times the mass difference between  $X$  and  $S$ , but not exceeding  $\frac{1}{2}$  of the digital range.

A sensitivity weight is not required if the electronic mass comparator that is used has been tested (with supporting data available) to determine that the balance has sufficient accuracy, resolution, repeatability, and stability so that no advantage is gained through the use of a sensitivity weight. For example, any possible errors must be less than what contributes to the uncertainty. When a mass comparator is used without a sensitivity weight, the sensitivity must be periodically verified and documented.

- 2.5.1.6. Determine which optional sequence will be used, A or B. Optional sequence A uses the standard on the balance for the first and fourth observations and the unknown on the balance for the second and third observations; this is often called the “SXXS” sequence. Optional sequence B starts with the unknown on the balance first and last with the standard on the balance for the second and third observations; this is often called the “XSSX” sequence. The primary advantage of sequence B is less handling of the mass standards. The advantage of sequence A is in the case where the unknown is a summation of weights that require careful arrangement on the balance pan only once.
- 2.5.1.7. Adjust the single pan balance or the combination balance so the first two readings of the double substitution fall in the first quarter of the optical scale or digital indications. The zero adjustment and tare adjustment may be used. Small weights may be placed on the balance pan to reach the desired reading range. These weights remain on the pan throughout the double

substitution. 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 (SXXS)

**Table 5. Optional Sequence A.**

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

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

- 2.5.2.1. 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.
- 2.5.2.2. 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  $O_2$ .
- 2.5.2.3. Observation 3. Add the sensitivity weight,  $sw$ , to the weights of observation 2. Release the pan, time the interval, and record observation  $O_3$ .
- 2.5.2.4. Observation 4. Remove weights  $X$  and  $t_x$  and replace with  $S$  and  $t_s$ . The sensitivity weight,  $sw$ , remains on the balance pan. Release the pan, time the interval, and record observation  $O_4$ .
- 2.5.2.5. Compare the two differences ( $O_2 - O_1$ ) and ( $O_3 - O_4$ ); they should not differ from one another by more than 2 standard deviations of the balance for this process and load. If this difference is exceeded, reject the data and redo the

measurements. Investigate possible causes of excess variability if measurements do not agree within these limits.

- 2.5.2.6. If repeated double substitutions are performed, the values between successive trials should not differ from one another by more than  $\pm 2$  standard deviations of the balance for this process and load. 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 (XSSX)

**Table 4. Optional Sequence B.**

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

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

## 3. Calculations

- 3.1. If no air buoyancy correction is performed, calculate the conventional mass correction,  $C_x$ , for the test weight as follows, according to the optional sequence used. Incorporate an uncorrected systematic uncertainty in the uncertainty calculations 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 no tare weights, and equal nominal values are used, those terms may all be deleted from the equations.

### 3.1.1. Optional Sequence A (SXXS)

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

### 3.1.2. Optional Sequence B (XSSX)

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

### 3.2. Mass Calculation with Air Buoyancy Correction

3.2.1. Calculate the air density,  $\rho_a$ , as described in the Appendix to SOP No. 2.

3.2.2. Calculate the mass  $M_x$  of the test weight, 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 (SXXS)

$$M_x = \frac{M_s \left(1 - \frac{\rho_a}{\rho_s}\right) + M_{t_s} \left(1 - \frac{\rho_a}{\rho_{t_s}}\right) - M_{t_x} \left(1 - \frac{\rho_a}{\rho_{t_x}}\right) + \left[ \frac{(O_2 - Q_1) + (O_3 - Q_4)}{2} \right] \left[ \frac{M_{sw} \left(1 - \frac{\rho_a}{\rho_{sw}}\right)}{O_3 - Q_2} \right]}{\left(1 - \frac{\rho_a}{\rho_x}\right)}$$

#### 3.2.2.2. Optional Sequence B (XSSX)

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

3.2.3. Calculate the 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<sup>3</sup> of  $X$ ,  $CM_x$ . It is recommended that the conventional mass be reported.

<sup>3</sup> 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<sup>3</sup>; reference temperature 20 °C; *normal* air density 0.0012 g/cm<sup>3</sup>. Conventional mass was formerly called “Apparent Mass versus 8.0 g/cm<sup>3</sup>” in the United States. See *OIML D28 (2004)*.

## 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 provided when requested by the customer for use when calibrating mechanical balances that have been adjusted to this reference density.

## 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 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 percent level of confidence. See SOP 29 for the complete standard operating procedure for calculating the uncertainty.

- 5.1. The expanded uncertainty for the standard,  $U$ , is obtained from the calibration 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 double 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 usually included at this calibration level include uncertainties associated with calculation of air density, 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 and 29 for additional details.
  - 5.5. 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 E617-97 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.
6. Report
- Report results as described in SOP No. 1, Preparation of Calibration/Test Reports.

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

**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*	Expanded Unc: From cal. report	Unc: k factor	Density $\text{g/cm}^3$
$S$					
$t_s$					
$X$					
$t_x$					
$S_c$					
$t_{S_c}$					
$sw$					

\*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:**

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

**Measurement Assurance (Duplication of the Process):**

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

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

**Laboratory data and conditions:**

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

**Mass standard(s) data:**

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

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

**Observations:**

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

**Measurement Assurance (Duplication of the Process):**

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

**Example: With Buoyancy Corrections**  
**Double Substitution Data Sheet**  
 (Optional Sequence A, SXXS)

**Laboratory data and conditions:**

Operator		HO	Before	After
Date	8/24/86	Temperature (°C)	22.3	22.2
Balance	M5SA	Pressure (mm Hg)	753.5	753.7
Load	10 g	Relative Humidity (%)	45	47
Standard deviation of the process, from control chart, $s_p$	0.002 9 mg	Degrees of Freedom		132

**Mass standard(s) data:**

ID	Nominal	Mass Correction*	Expanded Unc: From Cal. Rpt. (mg)	Unc: k factor	Density g/cm <sup>3</sup>
<u>Set 3</u> $S$	10 g	-0.679 mg	0.014 mg	3	8.00
$t_s$	---	---	---	---	---
<u>Set 432</u> $X$	10 g	TBD	TBD	2	7.84
$t_x$	---	---	---	---	---
<u>Set C</u> $S_c$	10 g	0.321 mg	0.025 mg	2	8.0
$t_{sc}$	---	---	---	---	---
<u>Set 3</u> $sw$	5 mg	-0.0227 mg	0.000 28	2	8.5

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

**Observations:**

Observation No.	Weights	Balance Observations, Units <u>mg</u>
Time: 8:35 AM		
1 ( $O_1$ )	$S + t_s$	1.268
2 ( $O_2$ )	$X + t_x$	1.821
3 ( $O_3$ )	$X + t_x + sw$	6.798
4 ( $O_4$ )	$S + t_s + sw$	6.245
Time: 8:47 AM		

**Measurement Assurance (Duplication of the Process):**

Observation No.	Weights	Balance Observations, Units <u>mg</u>
Time: 9:00 AM		
1 ( $O_1$ )	$S + t_s$	1.270
2 ( $O_2$ )	$S_c + t_{sc}$	2.271
3 ( $O_3$ )	$S_c + t_{sc} + sw$	7.248
4 ( $O_4$ )	$S + t_s + sw$	6.248
Time: 9:10 AM		
Check Standard in Control?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No	

Calculate the air density (SOP 2):

$$\rho_a = 1.1795 \text{ mg/cm}^3 = 0.0011795 \text{ g/cm}^3$$

Use equation 3.2.2.1 for optional sequence A (SXXS) with buoyancy corrections<sup>4</sup>:

$$M_x = \frac{M_s \left(1 - \frac{\rho_a}{\rho_s}\right) + M_{i_s} \left(1 - \frac{\rho_a}{\rho_{i_s}}\right) - M_x \left(1 - \frac{\rho_a}{\rho_x}\right) + \left[ \frac{(O_2 - Q) + (O_3 - Q)}{2} \right] \left[ \frac{M_{sw} \left(1 - \frac{\rho_a}{\rho_{sw}}\right)}{O_3 - Q} \right]}{\left(1 - \frac{\rho_a}{\rho_x}\right)}$$

$$M_x = \frac{9.999321 \left(1 - \frac{0.0011795}{8.0}\right) + 0 - 0 + \left[ \frac{(1.821 - 1.268) + (6.798 - 6.245)}{2} \right] \left[ \frac{0.0049773 \left(1 - \frac{0.0011795}{8.5}\right)}{6.798 - 1.821} \right]}{\left(1 - \frac{0.0011795}{7.84}\right)}$$

$$M_x = \frac{(9.9978461 + 0.000552957)}{0.99984949} = 9.9999041 \text{ g}$$

Calculate the mass (*true mass*) correction:

$$C_x = M_x - N_x$$

$$C_x = 9.9999041 \text{ g} - 10 \text{ g} = -0.0000959 \text{ g} = -0.0959 \text{ mg}$$

Calculate the conventional mass value:

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

$$CM_x = \frac{9.9999041 \left(1 - \frac{0.0012}{7.84}\right)}{0.999850}$$

<sup>4</sup> 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.

$$CM_x = \frac{9.9999041 (0.9998469)}{0.999850} = 9.99987351 \text{ g}$$

$$C_x = CM_x - N_x$$

$$C_x = 9.99987351 \text{ g} - 10 \text{ g}$$

$$C_x = -0.00012649 \text{ g} = -0.12649 \text{ mg}$$

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,  $U$ , must be divided by the k factor to determine the  $u_s$ .

$$u_c = \sqrt{(0.004667)^2 + (0.0029)^2 + (0.00000032)^2}$$

$$u_c = 0.0054946236 \text{ mg}$$

$$U = 0.0054946236 * 2 = 0.0109892473 \text{ mg}$$

### 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 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).

### Compliance Evaluation

We have to evaluate the correction with its expanded uncertainty to determine if the weight is in tolerance or not. 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 E617-97 and OIML R111.

Load = 10 g

ASTM E617		OIML R111	
Class	Tolerance (mg)	Class	Tolerance (mg)
3	0.25	F <sub>1</sub>	0.20
4	0.50	F <sub>2</sub>	0.60
5	2	M <sub>1</sub>	2.0

If we look at three times the uncertainty:  $0.011 \text{ mg} \times 3 = 0.033 \text{ mg}$ , we realize that the uncertainty complies with the 1/3 rule for the ASTM and OIML shown here.

Next, we look at the correction with the uncertainty:  $-0.126 \text{ mg} \pm 0.011 \text{ mg}$ .

We can see that the absolute value of the correction is within:  $0.115 \leq C_x \leq 0.137$ , therefore, it only complies with OIML class F<sub>1</sub>.

### Reporting

The conventional mass correction and uncertainty would be reported as follows:

$$C_x = -0.126 \text{ mg} \pm 0.011 \text{ mg}$$

**Example: Without Buoyancy Corrections**  
**Double Substitution Data Sheet**  
 (Optional Sequence B, XSSX)

**Laboratory data and conditions:**

Operator		HO	Before	After
Date	8/24/86	Temperature (°C)	22.3	22.3
Balance	CB 100	Pressure (mm Hg)	753.5	753.4
Load	30 g & 1 troy oz	Relative Humidity (%)	45	45
Standard deviation of the process, from control chart, $s_p$	0.018 mg	Degrees of Freedom	207	

**Mass standard(s) data:**

ID	Nominal	Mass Correction*	Expanded Unc: From cal. report	Unc: k factor	Density g/cm <sup>3</sup>
$S$	30 g	0.407 mg	0.022 mg	3	8
$t_s$	1.1 g	0.359 6 mg	0.006 3 mg	3	8.04
$X$	1 t oz	TBD	TBD	2	7.84
$t_x$	None	---	---	---	---
$S_c$	30 g	0.907 mg	0.030 mg	2	8
$t_{sc}$	None	---	---	---	---
$sw$	50 mg	-0.084 00 mg	0.000 65 mg	2	8.5

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

**Observations:**

Observation No.	Weights	Balance Observations, Units <u>mg</u>
Time: 9:00 AM		
1 ( $O_1$ )	$X + t_x$	20.93
2 ( $O_2$ )	$S + t_s$	17.21
3 ( $O_3$ )	$S + t_s + sw$	67.08
4 ( $O_4$ )	$X + t_x + sw$	70.81
Time: 9:05 AM		

**Measurement Assurance (Duplication of the Process):**

Observation No.	Weights	Balance Observations, Units
Time: 9:15 AM		
1 ( $O_1$ )	$S + t_s$	20.95
2 ( $O_2$ )	$S_c + t_{sc}$	21.45
3 ( $O_3$ )	$S_c + t_{sc} + sw$	71.32
4 ( $O_4$ )	$S + t_s + sw$	70.83
Time: 9:20 AM		
Check Standard in Control?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No	

Use equation 3.1.2 for optional sequence B (XSSX) with NO buoyancy corrections<sup>5</sup>:

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

Note: be careful to combine like units only!

$$C_x = 0.407 \text{ mg} + 1.1003596 \text{ g} - 0 + \left[ \frac{(20.93 - 17.21) + (70.81 - 67.08)}{2} \right] \left[ \frac{49.916 \text{ mg}}{67.08 - 17.21} \right] + 30 \text{ g} - 1 \text{ t oz}$$

$$C_x = 0.407 \text{ mg} + 1.1003596 \text{ g} - 0 + 3.7284359 \text{ mg} + 30 \text{ g} - 31.1034768 \text{ g}$$

$$C_x = 1104.495036 \text{ mg} - 1103.4768 \text{ g}$$

$$C_x = 1.018236 \text{ mg}$$

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,  $U$ , must be divided by the k factor for the standard and the tare weight to determine each  $u_s$ . The additional uncertainty for not performing the air buoyancy correction can be determined using the magnitude of the air buoyancy correction from SOP 2.

$$u_c = \sqrt{(0.00733)^2 + (0.0021)^2 + (0.018)^2 + (0.0016)^2}$$

$$u_c = 0.01961715 \text{ mg}$$

$$U = 0.01961715 * 2 = 0.039234 \text{ mg}$$

The conventional mass correction and uncertainty would be reported as follows:

$$C_x = 1.018 \text{ mg} \pm 0.039 \text{ mg}$$

OR

$$C_x = 0.0000327 \text{ t oz} \pm 0.0000013 \text{ t oz}$$

<sup>5</sup> 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.