

SOP No. 8

Recommended Standard Operations Procedure for Medium Accuracy Calibration of Mass Standards by Modified Substitution

1. Introduction

- 1.1. This SOP describes procedures to be followed for determining whether or not mass standards are within the tolerances specified for a particular class of standards (e.g., NIST Class F, ASTM Class 5, 6, 7 or OIML Classes M₁, M₂, and M₃) where the uncertainty is much smaller than the tolerance application. The described procedures permit the metrologist to report that the weights under test were compared against a reference standard with the results reported on the laboratory report form. The comparison is important because the built-in weights of a balance do not represent laboratory standards unless they have been formally calibrated. If a State law or other regulation requires that field weights be compared against the State (or reference) standards, this procedure can be used to fulfill this requirement. It is suitable for calibration when moderate accuracy is required, and 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. The expanded uncertainty with this procedure must be $\leq 1/3$ of the tolerance per ASTM and OIML standards. If uncertainty to tolerance ratios are greater than recommended, SOP 7 (single substitution) or SOP 4 (double substitution) is preferred.

Note: If you use SOP 8, you are most likely using working standards that are equivalent to your customer's weights. If this is the case, the working standards should be at a higher level of calibration than the weights being calibrated with them. Therefore, you will need a better balance and procedure to calibrate your working standards than the one used to calibrate your customer's weights.

1.2. Prerequisites

- 1.2.1. Verify that valid calibration values are available for the 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 standards should not be used at this level.
- 1.2.3. Verify that the balance that used 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 7, SOP 8, SOP 29, 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 (%)
III	18 °C to 25 °C, maximum change 1.0 °C/h	40 to 60 ± 10 / 4 h

2. Methodology

2.1. Scope, Precision, Accuracy

This method is applicable to all lower echelon mass calibration (tolerance testing) provided that the uncertainty requirements can be met. The achievable precision using this procedure is appropriate, provided the expanded uncertainty of the measurement is no more than one-third of the permissible tolerance of the mass standard tested. 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 mass to be tested is compared with a calibrated working standard by a modified substitution procedure. The comparison may be made using a single-pan, an equal-arm, or a fully-electronic balance. The reference standard is placed on the balance to obtain a convenient reference point and a sensitivity test is conducted. The error (departure from nominal value) of the weight tested is determined by comparing its reading to the reading obtained for the reference standard. A weight is considered to be within tolerance when the absolute value of its error plus its uncertainty does not exceed the tolerance established for the particular class of weight.

2.3. Apparatus/Equipment

2.3.1. Single-pan, equal arm, or full-electronic balance with sufficient capacity for the load tested and with readability equal to or less than one-tenth of the acceptable tolerance tested.

2.3.2. Mass standards calibrated with an expanded uncertainty of one-tenth or less than the tolerance tested. The calibration values must be traceable to NIST.

2.3.3. Calibrated sensitivity weights.

2.3.4. Uncalibrated counterweights, T , of approximately the same mass as the standard weights (for option C).

2.4. Procedure - Option A

Use of Single-Pan Balance

2.4.1. Select a reference standard of the same nominal value as the weight under test. Place the standard on the balance pan. Adjust the optical scale reading (See GMP No. 4) to approximately midscale using uncalibrated

tare weights and the balance's coarse and fine Zero controls. This setting must not be altered during a measurement sequence. Record the reading as O_1 .

- 2.4.2. Add a sensitivity weight equal to approximately one-quarter full scale reading and record reading as O_2 .
- 2.4.3. Calculate the value of a scale division using the equation in 3.2. If it is within $\pm 2\%$ of nominal value (usual case) the nominal value of a division can be used for tolerance testing.
- 2.4.4. Remove the sensitivity weight and adjust the optical scale to account for corrected value of standard used.

Example: Suppose that the nominal range of the optical scale is 100 mg and that the reference standard has a correction of -2.5 mg. The optical scale is adjusted to read 47.5 mg when the standard is on the pan. Under this condition, the reading 50.0 mg represents the nominal mass of the reference standard.

- 2.4.5. Remove the Standard.
- 2.4.6. Place the weight to be tested on the balance pan, read the optical scale and record reading as X_n . The error in the weight is the amount by which the indication deviates from the mid-scale reading. If the weight indication is more than the mid-scale value, the weight is heavy by the indicated difference; if the indication is less than the mid-scale value, the weight is light.
- 2.4.7. After several weights have been tested (no more than 10), put the standard on the balance pan and record the reading. The difference between this indication and the previous one for the standard indicates a balance drift. This drift will normally be very small. If the drift exceeds 10 % of the tolerance applicable to the weights under test or affects a measurement result to the extent that a weight may be out of tolerance, the measurement should be repeated and more frequent checks of the standard should be made or a more appropriate procedure should be used.
- 2.4.8. Readjust the optical scale at any time that a significant difference is observed when rechecking a standard.
- 2.4.9. Calculate the mass correction for the unknown weights using the appropriate equation in Section 3.

2.5. Procedure - Option A₁

Use of Single-Pan Balance

- 2.5.1. Select a reference standard of the same nominal value as the weight under test. Place the standard on the balance pan. Adjust the optical scale reading (See GMP No. 4) to midscale using uncalibrated tare weights and the balance's coarse and fine Zero controls. This setting must not be altered during a measurement sequence. Record the reading as O_1 .

- 2.5.2. Add a sensitivity weight equal to approximately one-quarter full scale reading and record reading as O_2 .
- 2.5.3. Calculate the value of a scale division using the equation in 3.2. If the sensitivity is within $\pm 2\%$ of nominal value (usual case) of the scale division, the nominal value of a division may be used.
- 2.5.4. Remove the sensitivity weight and re-adjust the optical scale to obtain a midscale indication, if the indication has changed from that set in 2.5.1.
- 2.5.5. Remove the Standard.
- 2.5.6. Place the weight to be tested on the balance pan, read the optical scale and record the indication as X_n . The error in the weight is the amount by which the indication deviates from the mid-scale reading. If the weight indication is more than the mid-scale value, the weight is heavier than the standard by the indicated difference; if the indication is less than the mid-scale value, the weight is lighter than the standard.
- 2.5.7. After several weights have been tested (no more than 10 without rechecking the standard) put the standard on the balance pan and record the reading. The difference between this indication and the previous one for the standard indicates a balance drift. This drift will normally be very small. If the drift exceeds 10 % of the tolerance applicable to the weights under test or affects a measurement result to the extent that a weight may be out of tolerance, the measurement should be repeated and more frequent checks of the standard should be made or a more appropriate procedure should be used.
- 2.5.8. Readjust the optical scale at any time that a significant difference is observed when rechecking a standard.
- 2.5.9. Calculate the correction of the unknown using the equation

$$C_x = C_s + (X_n - O_I).$$

2.6. Procedure - Option B

Use of Full Electronic Balance

- 2.6.1. Select a reference standard of the same nominal value as the weight under test. Place the standard on the pan. Zero the balance and record reading as O_I .
- 2.6.2. Add a calibrated sensitivity weight ($sw \geq 2$ times the tolerance but not exceeding 1 % of the balance capacity) and record the reading as O_2 . Verify whether the nominal scale division is within $\pm 2\%$ of nominal value of the scale division using the equation in 3.2. If so, the nominal value of the scale division may be used.
- 2.6.3. Remove sensitivity weight and zero the balance so weight differences, d , can be read directly from the balance indications.

- 2.6.4. Remove all weights from the balance pan.
- 2.6.5. Place the weight to be tested on the balance pan. Record the reading as X_n .
- 2.6.6. After several weights have been tested (no more than 10 without rechecking the standard) recheck the zero as in 2.5.3 and record the reading. The difference between this indication and the previous one for the standard indicates a balance drift. This drift will normally be small. If the drift exceeds 10 % of the tolerance applicable to the weights under test or affects a measurement result to the extent that a weight may be out of tolerance, the measurement should be repeated and more frequent checks of the standard should be made or a more appropriate procedure should be used.
- 2.6.7. Readjust the zero at any time that a significant difference is observed when rechecking a standard.
- 2.6.8. Calculate the mass correction for each weight using the equation

$$C_x = C_s + (X_n - O_I) = C_s + X_n \text{ (when } O_I \text{ is zeroed)}$$

2.7. Procedure - Option C

Use of Equal Arm Balance

- 2.7.1. Select a reference standard of the same nominal value as the weight under test. Place the standard on the left balance pan together with small, calibrated weights equal to the correction required for the standard, provided it is light. If (and only if) the standard is heavy, do nothing further at this point but follow instructions in 2.6.4. Add sufficient counterweights to the right pan to obtain a sum of turning points of approximately twice midscale value. If necessary, number the graduated scale such that adding weights to the left pan will increase the balance reading. Record the sum of the turning points as θ_1 .
- 2.7.2. Add an appropriate calibrated sensitivity weight to the left pan and record the sum of the turning points as θ_2 . Calculate the sensitivity,

$$\text{sensitivity} = \frac{CM_{sw}}{(\theta_2 - \theta_1)}$$

where CM_{sw} is the conventional mass of the sensitivity weight.

- 2.7.3. Remove all weights from the left pan.
- 2.7.4. Place weight to be tested on the left pan. If the standard used in 2.6.1 was heavy, add small correction weights to the left pan, equivalent to the correction required for the standard. Add small, calibrated tare weights as required to left or right pan to obtain an approximate balance and record the sum of the turning points as X_n .

2.7.5. Calculate the mass correction of the weight tested as follows.

2.7.5.1. If added tare weights are placed on the left pan (t_x).

$$C_x = (X_n - O_1) \left[\frac{CM_{sw}}{(O_2 - O_1)} \right] - CM_{t_x}$$

2.7.5.2. If added tare weights are placed on the right pan (t_s)

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

2.7.6. After several weights have been tested (no more than 10 without rechecking the standard), recheck the turning point θ_I , as described in 2.6.1. Only a small difference should be observed. If the difference exceeds 2 % of the sum of the turning points in O_I , the measurement should be repeated and more frequent checks of the standard should be made or a more appropriate procedure should be used.

2.8. Tolerance Evaluation

2.8.1. Compare the correction plus the expanded uncertainty of the weight tested with the tolerance for the class of weights to which it belongs. If the absolute value of the correction plus the expanded uncertainty is numerically smaller than the tolerance, the weight is considered to be within tolerance. If the correction is larger than permissible, the weight is considered to be outside the tolerance and appropriate action should be taken. It is recommended that weights whose absolute value of the correction exceeds 75 % of the tolerance limit be adjusted.

Table 2. Example of Weighing Sequence

Measurement No.	Weights on Pan	Observation
1	S	O_1
2	$S + sw$	O_2
3	S	O_3
4	X_1	O_4
5	X_2	O_5
6	X_3	O_6
7	X_4	O_7
8	X_5	O_8
9	X_6	O_9
10	X_7	O_{10}
11	X_8	O_{11}
12	X_9	O_{12}
13	S_c	O_{13}
14	S	O_{14}

3. Calculations

- 3.1. Air buoyancy corrections are generally *not* made with the modified substitution. 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) are included.

Table 3. Symbols used

Symbol	Description
CM_i	conventional mass of weight i
N_s	nominal value of S
N_x	nominal value of X

- 3.2. Evaluate the sensitivity of the balance:

$$sensitivity = \frac{CM_{sw}}{(O_2 - O_1)}$$

If the sensitivity error is less than 2 % of the nominal value of a division on the optical scale or the electronic range of operation, proceed with the modified substitution. If the sensitivity error is greater, SOP 7, Single Substitution may be acceptable.

- 3.3. Calculate the mass correction of each unknown weight as follows if the correction for the standard IS NOT used in setting a reference point on the balance:

$$C_x = C_s + d$$

$$C_x = C_s + (X_n - O_1)$$

Note: If an electronic balance is used and zeroed with the standard on the balance, O_I is "0" and d becomes the X_n balance reading.

- 3.4. Calculate the mass correction of each unknown weight as follows if the correction for the standard IS used in setting a nominal reference point on the balance:

$$C_x = (X_{\text{balance reading}} - N_{\text{nominal reference point}})$$

Note: In this case the standard and its correction are used to artificially *set* a perfect nominal reference point for use in comparing the unknown weights.

- 3.5. If tare weights and unequal nominal values are used, use the following equation for modifying section 3.3:

$$C_x = C_s + CM_{ts} - CM_{tx} + (X_n - O_I) + N_s - N_x$$

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. Standard deviation of the measurement process from control chart performance (See SOP No. 9.) The value for s_p is obtained from the control chart data for check standards using modified substitution measurements.
- 5.3. Other standard uncertainties usually included at this calibration level include uncertainties associated with lack of performing buoyancy corrections and can be determined using the equation given in SOP 2 for the magnitude of the air buoyancy correction. Buoyancy corrections are not generally needed at the uncertainty level this procedure is designed for.

5.4. The expanded uncertainty, U , must be $\leq 1/3$ of the tolerance applicable as per ASTM E617-97 and OIML R111.

6. Report

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

Appendix Modified Substitution Data Sheet

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*	Expanded Unc: From cal. Report	Unc: k factor	Density g/cm ³
S					
X					
sw					
t_x					
t_s					

*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		
2 (O_2)	$S + sw$		
Error < 2 % of optical scale or electronic range? Yes No			
3 (O_3)	S		
4 (O_4)	X_1		
5 (O_5)	X_2		
6 (O_6)	X_3		
7 (O_7)	S_c		
8 (O_8)	S		
Time:	Drift < 1/10 Tol. ? : Yes No		

Up to 10 unknown weights may be checked with this procedure if the drift is less than 1/10 of the tolerance.

Appendix

Modified Substitution Data Sheet

Laboratory data and conditions:

Operator	GH		
Date	9/30/96	Temperature	22.5 °C
Balance	PM 2000 MC (2300 g)	Pressure	747.5 mmHg
Load	5 lb	Relative Humidity	45 %
Standard deviation of the process, from control chart, s_p			0.85 mg

Mass standard(s) data:

ID	Nominal	Mass Correction	Expanded Unc: From cal. Report	Unc: k factor	Density g/cm ³
S	5 lb	7.5 mg	0.50 mg	3	7.85
X	5 lb				7.85
sw	5 g	0.000 002 g			
t_x	None				
t_s	None				
S_c	5 lb	2.4 mg	0.73 mg	2	7.95

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

Observations:

Observation No.	Weights	Balance Observations, Units 0.001 g
Time: 11:00 am		
1 (O_1)	S	0000
2 (O_2)	$S + sw$	4999
Error < 2 % of optical scale or electronic range? Yes No		
3 (O_3)	S	0000
4 (O_4)	X_1	0205
5 (O_5)	X_2	0104
6 (O_6)	X_3	- 0089
7 (O_7)	S_c	- 0005
8 (O_8)	S	0003
Time: 11:20 am		Drift < 1/10 Tol. ? : Yes No

Up to 10 unknown weights may be checked with this procedure if the drift is less than 1/10 of the tolerance.

Sensitivity guidelines indicate a sensitivity weight, greater than or equal to 2 times the tolerance (460 mg) but not exceeding 1 % of the balance capacity (23 g). For a 5 lb load, the Class F tolerance is 230 mg so a 5 g weight was selected.

A 2 % sensitivity error in a 5 gram range would allow readings between 4.900 g and 5.100 g in the sensitivity test. Calculate the sensitivity:

$$\text{sensitivity} = \frac{5.000\,002\text{ g}}{4.999\text{ div}} = 1.000\,200\,4\text{ g/div}$$

which deviates less than 2 % of the nominal value of a division.

The drift observed was 3 mg and is less than 1/10 of the tolerance and 23 mg could be allowed, so the error for each nominal weight may be calculated as follows.

$$C_{x_1} = 7.5\text{ mg} + (205\text{ mg} - 0) = 212.5\text{ mg}$$

$$C_{x_2} = 7.5\text{ mg} + (104\text{ mg} - 0) = 111.5\text{ mg}$$

$$C_{x_3} = 7.5\text{ mg} + (-89\text{ mg} - 0) = -81.5\text{ mg}$$

$$C_{Sc} = 7.5\text{ mg} + (-5\text{ mg} - 0) = 2.5\text{ mg}$$

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 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. Since the working standard was previously calibrated with buoyancy corrections and is the same density as the unknown weights, the uncertainty for the buoyancy correction drops out of the equation.

$$u_c = \sqrt{(0.167)^2 + (0.85)^2}$$

$$u_c = 0.866\,19\text{ mg}$$

$$U = 0.866\,19 * 2 = 1.732\,38\text{ mg}$$

The expanded uncertainty is less than 1/3 of the tolerance (76 mg) so the unknown weights can either be reported as “within tolerance” or the values and uncertainties can be reported. The value plus the uncertainty for the first unknown X_1 is within 25 % of the Class F tolerance, therefore it is desirable to adjust the weight closer to nominal value, although based on the tolerances and uncertainties, and it can be claimed as “within tolerance.”