

## SOP No. 3

### Recommended Standard Operations Procedure

for

### Weighing by Double Substitution

### Using an Equal-Arm Balance

#### 1. Introduction

##### 1.1. Purpose

The double substitution procedure is one in which a standard and an unknown weight are intercompared twice to determine the average difference between the two weighings. Any effects of inequality of arms and linear drift are eliminated by the weighing sequence used. Accordingly, the procedure is especially useful for high accuracy calibrations.

##### 1.2. Prerequisites

- 1.2.1. Mass standards must be available with calibration certificates traceable to NBS.
- 1.2.2. The balance used must be in good operating condition as verified by a valid control chart or preliminary experiments to ascertain its performance quality.
- 1.2.3. The operator must be experienced in precision weighing techniques.

#### 2. Methodology

##### 2.1. Scope, Precision, Accuracy

This method is applicable to all weighings utilizing an equal arm balance. Because considerable effort is involved, it is most useful for calibrations of the highest accuracy. The precision will depend upon the sensitivity of the balance and the care exercised in making the required weighings. The accuracy will depend on the accuracy of calibration of the standard weights and the precision of the intercomparison.

##### 2.2. Summary

The standard is balanced with respect to a counterweight. The standard is then replaced by the test weight which is weighed with respect to the same counterweight. A small 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 test weights as well as a determination of the sensitivity of the balance under the load conditions. All weighings are made at regularly spaced time intervals to average out any effects due to linear instrument drift.

### 2.3. Apparatus/Equipment

- 2.3.1. Precision equal-arm balance with sufficient capacity and sensitivity for the calibrations planned. The index scale of the balance is conveniently numbered from 0 to 20 with 10 as the center division, although other numbering systems such as 0 to 200 are possible. A system in which the center division is 0 is not recommended since the negative readings that result can cause observational and/or computational problems. The graduations are so numbered that the addition of a small weight to the left arm will increase the scale reading.
- 2.3.2. Standard weights with valid calibrations, traceable to NBS. The sensitivity weight may be a calibrated secondary standard. The use of secondary standards as sensitivity weights reduces wear on the primary mass standards.
- 2.3.3. Counterweights, (uncalibrated), of approximately the same mass as the standard weights. Lead shot in a suitable container is useful for this purpose.
- 2.3.4. Small calibration weights (usually decimal fractions) to be used as tare weights.
- 2.3.5. Equipment capable of loading and unloading weights on the balance without damage to either (especially important in the case of large weights).
- 2.3.6. Stop watch or other timing device to observe time of each trial measurement.

### 2.4. Symbols

The following symbols are used in this procedure:

- S - standard weight
- X - weight calibrated
- T - counterweight
- t - small calibrated 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 of a specific weight. Subscripts s, x, t, sw, are used to identify the weight
- AM - the apparent mass of a specific weight. Subscripts s, x, t, sw, are used to identify the weight.

## 2.5. Procedure

### 2.5.1. Preliminary Procedure

Conduct preliminary measurements (without recording data) to determine the values for  $T$ ,  $t_s$ ,  $t_x$ , and  $sw$  for use in measurement procedures, 2.5.2, or 2.5.3. This will serve to warm up the balance and facilitate the actual measurements in which the trials should be observed at regular time intervals. Estimate the time required to complete a trial (see 2.5.2) and maintain essentially equal intervals between successive trials (see 2.5.2.5). In a series of calibrations, this preliminary procedure is only required when new values need to be determined for  $T$ ,  $t_s$ ,  $t_x$ , and  $sw$ .

### 2.5.2. Measurement Procedure, Option A

Measurement No.	Left Pan (arm)	Right Pan (arm)	Observation
1	$S + t_s$	$T$	$O_1$
2	$X + t_x$	$T$	$O_2$
3	$X + t_x + sw$	$T$	$O_3$
4	$S + t_s + sw$	$T$	$O_4$

The turning points,  $O_1$ ,  $O_2$ ,  $O_3$ , and  $O_4$  are observed as described in GMP No. 1. All observations should be recorded on suitable data sheets, such as those in the Appendix.

2.5.2.1. Observation 1. Place the standard weight(s),  $S$ , equivalent to the nominal weight of the test weight, on the left pan (arm) of the balance and add a slight excess of counter weight ( $T$ ) to the right pan (arm). Add tare weight,  $t_s$ , to the left pan (arm) to obtain an approximate balance. Record the sum of the turning points and the time.

2.5.2.2. Observation 2. Remove weight(s)  $S$  and replace with test weight,  $X$ . Adjust tare weight,  $t_x$ , to obtain an approximate balance condition within one division of the sum of turning points obtained for  $O_1$  (for a 0 to 20 division graduated scale). (If  $S$  and  $X$  are approximately equal,  $t_x$  may equal  $t_s$ .) Record the sum of the turning points and the time.

2.5.2.3. Observation 3. Add a small sensitivity weight,  $sw$ , to the weights of observation 2. The value of  $sw$  should be such as to change the turning points by about 4 divisions on a 0 to 20 scale (40 divisions on a 0 to 200 scale). Record the sum of the turning points and the time.

2.5.2.4. Observation 4. Remove weight X and  $t_x$  and replace with S and  $t_s$ . Weight sw remains on the balance pan. Record the sum of the turning points and the time.

2.5.2.5. Calculate the time intervals between the successive trials. These should not differ from one another by more than  $\pm 20\%$ . If this difference is exceeded, reject the data and take a new series of measurements that will so agree.

### 2.5.3. Measurement Procedure, Option B

Measurement No.	Left Pan (arm)	Right Pan (arm)	Observation
1	X + $t_x$	T	O <sub>1</sub>
2	S + $t_s$	T	O <sub>2</sub>
3	S + $t_s$ + sw	T	O <sub>3</sub>
4	X + $t_x$ + sw	T	O <sub>4</sub>

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

## 3. Calculations

3.1. Calculate the sums of the turning points, O<sub>1</sub>, O<sub>2</sub>, O<sub>3</sub> and O<sub>4</sub> (Ref. GMP No. 1).

3.2. Calculate the correction, C<sub>x</sub>, required for the test weight, as follows, according to the optional sequence used. In each case, C<sub>s</sub> is the apparent mass correction required for the standard weight used, including that for the tare weights as appropriate.

3.2.1. No air buoyancy correction. Calculate the apparent mass correction, C<sub>x</sub>, for the test weight as follows, according to the optional sequence used. In each case, the appropriate apparent mass corrections for the standard weight(s), C<sub>s</sub>, the tare weights AM<sub>t<sub>s</sub></sub> and AM<sub>t<sub>x</sub></sub>, and the sensitivity weights, AM<sub>sw</sub>, are included. The symbols N<sub>s</sub> and N<sub>x</sub> refer to the nominal values of S and X, respectively.

#### 3.2.1.1. Optional Sequence A

$$C_x = C_s + AM_{t_s} - AM_{t_x} + \frac{(O_2 - O_1 + O_3 - O_4)}{2} \frac{(AM_{sw})}{(O_3 - O_2)} + N_s - N_x$$

#### 3.2.1.2. Optional Sequence B

$$C_x = C_s + AM_{t_s} - AM_{t_x} + \frac{(O_1 - O_2 + O_4 - O_3)}{2} \frac{(AM_{sw})}{(O_3 - O_2)} + N_s - N_x$$

### 3.2.2. Air Buoyancy Correction

3.2.2.1. Calculate the air density,  $\rho_A$ , as described in section 8 of the Appendix to SOP No. 2 or obtain from Table 9.9.

3.2.2.2. Calculate the mass  $M_x$  of the test weight 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.

#### 3.2.2.2.1. Optional Sequence A

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

#### 3.2.2.2.2. Optional Sequence B

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

3.2.2.3. Calculate the mass correction  $C'_x$ , as follows:

$$C'_x = M_x - N_x$$

where  $N_x$  is the nominal value for X.

3.2.2.4. Calculate the apparent mass of X ( $AM_x$ ), versus the desired reference density of 8.0 g/cm<sup>3</sup> or brass. It is recommended that the apparent mass versus 8.0 g/cm<sup>3</sup> be reported unless otherwise requested. The density,  $\rho_x$ , must be in g/cm<sup>3</sup>.

3.2.2.4.1. Apparent mass versus 8.0 g/cm<sup>3</sup>

$$AM_x \text{ vs } 8.0 = M_x \frac{\left(1 - \frac{.0012}{\rho_x}\right)}{0.999850}$$

### 3.2.2.4.2. Apparent mass versus brass

$$AM_x \text{ vs brass} = M_x \frac{1 - \frac{.0012}{\rho_x}}{.999857}$$

## 4. Assignment of Uncertainty

The limits of uncertainty, U, include estimates of the uncertainty of the mass standards used,  $U_s$ , plus the uncertainty of measurement,  $U_m$ , at the 99.73% level of confidence. The latter is estimated by

$ts$

where  $s$  is the standard deviation of measurement and  $t$  is obtained from Table 9.3.

Then 
$$U = \pm [U_s + ts]$$

### 4.1. Precision of Measurement Known from Control Chart Performance. (See SOP No. 9.)

The value for  $s$  is obtained from the control chart limits and current knowledge that the measurements are in a state of statistical control. This will need to be ascertained by measurement of at least one check standard while the above measurements are in progress.

Use the value of  $t$  (corresponding to a probability level of 99.73%) from Table 9.3 appropriate for the number of degrees of freedom,  $\nu$ , on which the control limits of the control chart are based.

### 4.2. Precision Estimated from Series of Measurements

Measure a stable test object at least 7 times, no two measurements of which may be made on a single day. Calculate the mean and the standard deviation in the conventional manner. The latter is the value of  $s$  that is used in Section 4.1. In this case select the value for  $t$  from Table 9.3 based on the number of degrees of freedom involved in computing  $s$ .

Note: Repetitive measurements made on the same day estimate only the short-term standard deviation.

## 5. Report

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

**Appendix**

**Double Substitution Data Sheet  
for Equal-Arm Balance (Option A)**

Test No.: \_\_\_\_\_ Sheet No.: \_\_\_\_\_ Date: \_\_\_\_\_

Item Identification: \_\_\_\_\_ Balance: \_\_\_\_\_

Standard Identification: \_\_\_\_\_ Observer: \_\_\_\_\_

Temperature: \_\_\_\_\_ Pressure: \_\_\_\_\_ Rel. Hum.: \_\_\_\_\_

$C_s(C'_s) =$  \_\_\_\_\_  $\pm$  \_\_\_\_\_  $AM_{sw}(M_{sw}) =$  \_\_\_\_\_

$\rho_s =$  \_\_\_\_\_  $\rho_x =$  \_\_\_\_\_  $\rho_{sw} =$  \_\_\_\_\_

$AM_{t_s}(M_{t_s}) =$  \_\_\_\_\_  $\pm$  \_\_\_\_\_  $AM_{t_x}(M_{t_x}) =$  \_\_\_\_\_  $\pm$  \_\_\_\_\_

$\rho_{t_s} =$  \_\_\_\_\_  $\rho_{t_x} =$  \_\_\_\_\_

Time \_\_\_\_\_ Balance standard deviation = \_\_\_\_\_

Measurement No.	Weights	Turning Points		
	Left Arm	Low	High	Sum
1	$S + t_s$			$O_1 -$
2	$X + t_x$			$O_2 -$
3	$X + t_x + sw$			$O_3 -$
4	$S + t_s + sw$			$O_4 -$

Time \_\_\_\_\_

Appendix

Double Substitution Data Sheet  
for Equal-Arm Balance (Option B)

Test No.: \_\_\_\_\_ Sheet No.: \_\_\_\_\_ Date: \_\_\_\_\_

Item Identification: \_\_\_\_\_ Balance: \_\_\_\_\_

Standard Identification: \_\_\_\_\_ Observer: \_\_\_\_\_

Temperature: \_\_\_\_\_ Pressure: \_\_\_\_\_ Rel. Hum.: \_\_\_\_\_

$C_s(C'_s) =$  \_\_\_\_\_  $\pm$  \_\_\_\_\_  $AM_{sw}(M_{sw}) =$  \_\_\_\_\_

$\rho_s =$  \_\_\_\_\_  $\rho_x =$  \_\_\_\_\_  $\rho_{sw} =$  \_\_\_\_\_

$AM_{t_s}(M_{t_s}) =$  \_\_\_\_\_  $\pm$  \_\_\_\_\_  $AM_{t_x}(M_{t_x}) =$  \_\_\_\_\_  $\pm$  \_\_\_\_\_

$\rho_{t_s} =$  \_\_\_\_\_  $\rho_{t_x} =$  \_\_\_\_\_

Time \_\_\_\_\_ Balance standard deviation = \_\_\_\_\_

Measurement No.	Weights	Turning Points		
	Left Arm	Low	High	Sum
1	$X + t_x$			$O_1 -$
2	$S + t_s$			$O_2 -$
3	$S + t_s + sw$			$O_3 -$
4	$X + t_x + sw$			$O_4 -$

Time \_\_\_\_\_

Appendix

Double Substitution Data Sheet  
for Equal-Arm Balance (Option A)

Test No.: 137 Sheet No.: 1 Date: 8/27/86

Item Identification: 1000 lb No. 623 Balance: Russell

Standard Identification: #11 and #22 Observer: HO

Temperature: 23.5°C Pressure: 746.1 mm Hg Rel. Hum.: 45%

$\sigma_s(C'_s) = \underline{0.0047 \text{ lb}} \pm \underline{.0030 \text{ lb}}$   $\Delta M_{sw}(M_{sw}) = \underline{0.01 \text{ lb}}$

$\rho_s = \underline{8.0 \text{ g/cm}^3}$   $\rho_x = \underline{7.0 \text{ g/cm}^3}$   $\rho_{sw} = \underline{7.8 \text{ g/cm}^3}$

$\Delta M_t(M_{t_s}) = \underline{3.625} \pm \underline{.000075 \text{ lb}}$   $\Delta M_t(M_{t_x}) = \underline{3.642 \text{ lb}} \pm \underline{\text{negligible}}$

$\rho_{t_s} = \underline{8.0 \text{ g/cm}^3}$   $\rho_{t_x} = \underline{7.8 \text{ g/cm}^3}$

Time 10:45 a Balance standard deviation = 0.0018 lb

Measurement No.	Weights	Turning Points		
	Left Arm	Low	High	Sum
1	S + t <sub>s</sub>	<del>5.1</del> 5.2	<del>15.3</del> 15.2 15.2	O <sub>1</sub> = 20.4
2	X + t <sub>x</sub>	<del>5.8</del> 5.9 5.9	<del>14.1</del> 14.1	O <sub>2</sub> = 20.0
3	X + t <sub>x</sub> + sw	<del>6.1</del> 6.2	<del>17.6</del> 17.5 17.4	O <sub>3</sub> = 23.65
4	S + t <sub>s</sub> + sw	<del>6.3</del> 6.4 6.4	<del>16.3</del> 16.2	O <sub>4</sub> = 22.6

Time 11:05 a

$$\rho_a = 1.163 \text{ mg/cm}^3 = 0.001163 \text{ g/cm}^3$$

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

$$M_x = \left[ 1000.0047 \left(1 - \frac{.001163}{8.0}\right) + 3.625 \left(1 - \frac{.001163}{8.0}\right) - 3.642 \left(1 - \frac{.001163}{7.8}\right) + \frac{20.0 - 20.4 + 23.65 - 22.6}{2} \cdot \frac{.01 \left(1 - \frac{.001163}{7.8}\right)}{23.65 - 20.0} \right] \left/ \left(1 - \frac{.001163}{7.0}\right)\right.$$

$$M_x = \frac{999.8593243 + 3.6244730 - 3.6414570 + .0008903}{.99983386}$$

$$M_x = 1000.009372 \text{ lb}$$

$$\text{Uncertainty} = t_s + U_s \quad \text{Degrees of freedom in } s = 20$$

$$\text{Uncertainty} = 3.422 (.0018) + (.0030 + .000075)$$

$$\text{Uncertainty} = 0.0092346 \text{ lb}$$

$$M_x = 1000.0094 \pm 0.0092 \text{ lb}$$

$$C'_x = M_x - N_x = 0.0094 \pm 0.0092 \text{ lb}$$

$$AM_x \text{ vs } 8.0 = M_x \frac{\left(1 - \frac{.0012}{\rho_x}\right)}{0.999850}$$

$$AM_x \text{ vs } 8.0 = 1000.0094 \frac{\left(1 - \frac{.0012}{7.0}\right)}{.999850}$$

$$AM_x \text{ vs } 8.0 = 999.9880 \pm .0092 \text{ lb}$$

$$C_x = AM_x \text{ vs } 8.0 - N_x$$

$$C_x = 999.9880 - 1000 = -.0120 \pm .0092 \text{ lb}$$