

AQS110 – Introduction to Quality and Metrology – Fall 2016
LABORATORY EXERCISE #5

CALIBRATION

Purpose

The purpose of this laboratory exercise is to demonstrate how to use various tools for verifying (calibrating) equipment.

The lab will be conducted in one day; however the data collected will be used for a variety of future exercises

Format

Students will be working in groups of 2 or 3. Individual work stations containing the following equipment have been set-up.

- Top loading balance
- Analytical Balance
- Various volumetric pipettes
- Various micropipettes
- Calibrated weight set.
- 1 L flask with temperature equilibrated water
- 1 thermometer

Each station will also have the data collection sheets needed for recording the measurement results for the individual parts.

Background

As described in Laboratory #4, measurement is a method for evaluating a property or characteristic of an object and describing it with a numerical or nominal value.

Applied/Industrial metrology applies measurement science to manufacturing and other processes. This is often used by the company to determine whether or not to ship the product. Applied metrology is twofold:

- a) it ensures the suitability of the measurement instruments, their calibration and the quality control of the measurements; and
- b) maintains traceability of the calibration for the instruments to ensure confidence in the results.

An industrial metrology program typically has two aspects: measurement fundamentals and calibration. We will be exploring calibration in this exercise.

Due Date: October 19, 2016 for data (pages 5 & 6) and post-lab question responses (page 7 & 8).

Laboratory Exercise:

The calibrated weight standards will be used to verify that the balances are level and operating properly. Two types of balances will be used in this exercise: a) top loading (no doors) and b) analytical (with doors).

- Top loading balances are exposed to the environment (drafts). Their sensitivity will be limited. This balance needs to be level, the pan and top of the balance needs to be clean.
- Analytical balances do have some environmental shielding. Their sensitivity is greater than the top loading, but only when the doors are closed. This balance also needs to be level, the pan and interior chamber need to be clean.

Once standardized, the balances will then be used to verify that the volumetric pipettes and micropipettes are delivering the appropriate volumes.

- The volumetric pipettes are designed to deliver and should be allowed to drain using gravity; there may be some liquid remaining in the tip.
- The micropipettes are also designed to fill and deliver the expected volume by depressing the button at the top of the pipette; there should be no liquid remaining in the tip when delivery is complete.

Water will be the solvent used for this experiment as its density (1.00 g/ml) allows for direct conversion from grams to mL; that is 1 mL will weigh 1.00 g and 1 μ L will weigh 0.001 g (1 mg). The table below lists more accurate densities based on temperatures.

Temperature		Water Density (g/mL)
$^{\circ}$ C	$^{\circ}$ F	
20	68	0.99821
22	72	0.99777
25	77	0.99705

Useful conversions

$$\begin{array}{ll} 1 \text{ g} = 1000 \text{ mg} & 1 \text{ mg} = 0.001 \text{ g} \\ 1 \text{ mL} = 1000 \text{ } \mu\text{L} & 1 \text{ } \mu\text{L} = 0.001 \text{ mL} \end{array}$$

Converting weight of water to volume, notice the number of significant digits:

@ 22 $^{\circ}$ C 1.233 g of water =?? mL this will be determined based on the sensitivity of the scale

$$1.23 \text{ g} \times \frac{\text{mL}}{1.00 \text{ g}} = 1.23 \text{ mL}$$

$$0.123 \text{ g} \times \frac{\text{mL}}{0.998 \text{ g}} = 0.123 \text{ mL (1230 } \mu\text{L)}$$

$$1.233 \text{ g} \times \frac{\text{mL}}{0.998 \text{ g}} = 1.235 \text{ mL}$$

$$0.1235 \text{ g} \times \frac{\text{mL}}{0.9978 \text{ g}} = 0.1237 \text{ mL (1237 } \mu\text{L)}$$

$$1.2335 \text{ g} \times \frac{\text{mL}}{0.9978 \text{ g}} = 1.2362 \text{ mL}$$

$$0.12356 \text{ g} \times \frac{\text{mL}}{0.99777 \text{ g}} = 0.12384 \text{ mL (1238 } \mu\text{L)}$$

Procedure:**1. General Instructions**

- 1.1. Verify that balance is level and clean. If cleaning is needed, use a kimwipe® dampened with 70:30 alcohol:water.
- 1.2. Weight standards shall be handled with gloves on or the forceps only. Handling the weights with your bare hands can leave skin oils behind which can affect the results.
- 1.3. Before taking a measurement ensure the balance has been zeroed, by using the tare button.
- 1.4. Record the weights, including all the numbers beyond the decimal point that are displayed.
- 1.5. The forms located on pages 5 and 6 shall be treated as quality records, GDP rules apply.

2. Verifying / Calibrating Volumetric Pipettes**2.1. Calibrating the top-loading balance**

Note: each measurement will be taken three times, the standard shall be removed from the pan and the scale re-zeroed between measurements.

- 2.1.1. Record the standard weight set serial number, balance ID number and balance sensitivity onto the form (page 5).
- 2.1.2. Ensure the balance has been zeroed prior to starting.
- 2.1.3. Beginning with the smallest weight and using forceps, place the standard on the pan
- 2.1.4. Allow the weight to stabilize.
- 2.1.5. Record the weight on the form provided.
- 2.1.6. Repeat steps 2.1.2 and 2.1.3 two more times, verifying the balance begins at zero.
- 2.1.7. Sign and date the results.
- 2.1.8. Using the next weight, place the standard on the pan, allow to stabilize, record the weight and repeat two more times.

2.2. Verifying the pipette volume

Note: Three weights per pipette will be taken, after recording the weight of the water delivered, tare (zero) the scale. The beaker will remain on the pan, but the display will be changed to zero.

- 2.2.1. Record the room temperature as displayed on the room thermostat.
- 2.2.2. Using the thermometer provided, determine and record the temperature of the water contained in the 1L flask.
- 2.2.3. The 600 mL beaker will be used for verifying the 10 mL and 20 mL pipettes.
- 2.2.4. The 100 mL beaker will be used for verifying the 2 mL and 5 mL pipettes.
- 2.2.5. With the pan empty, tare the balance to ensure it begins at zero.
- 2.2.6. Place the 100 mL beaker on the pan and press tare; 0.00 should appear in the display.
- 2.2.7. Beginning with the 2 mL pipette draw up the appropriate amount of water. Be sure to check the meniscus at eye level.
- 2.2.8. Dispense the water from the pipette into the beaker that is on the scale pan. Allow the display to stabilize and record the weight.
- 2.2.9. Tare the balance and repeat dispensing the pipette volume into the beaker, taring between each measurement.
- 2.2.10. Repeat steps 2.2.8 and 2.2.9 for the 5 mL pipette.
- 2.2.11. When the weights for the 2 mL and 5 mL pipettes have been completed, remove the beaker and re-zero the balance, with nothing on the pan.

- 2.2.12. Place the 600 mL beaker on the pan and press tare; 0.00 should appear in the display.
- 2.2.13. Repeat the “dispense and measure” process (2.2.7-2.2.10) for the 10 mL and 20 mL pipettes.

3. Verifying / Calibrating Micropipettes

3.1. Calibrating the analytical balance

Note: each measurement will be taken three times, the standard shall be removed from the pan and the scale re-zeroed between measurements.

- 3.1.1. Record the standard weight set serial number, balance ID number and balance sensitivity onto the form (page 6).
- 3.1.2. Ensure the balance has been zeroed prior to starting.
- 3.1.3. Beginning with the smallest weight and using forceps, place the standard on the pan
- 3.1.4. Allow the weight to stabilize.
- 3.1.5. Record the weight on the form provided.
Note: Be careful when handling the smallest weights, these are easily damaged. Use the forceps rather than handling them directly. Be sure to place them back into the correct storage location when complete.
- 3.1.6. Repeat steps 3.1.2 and 3.1.3 two more times, verifying the balance begins at zero.
- 3.1.7. Sign and date the results.
- 3.1.8. Using the next weight, place the standard on the pan, allow to stabilize, record the weight and repeat two more times.

3.2. Verifying the micropipette volume

Note: Three weights per volume will be taken, after recording the weight of the water delivered, the scale shall be tared (zeroed). The container will remain on the pan, but the display will be changed to zero.

- 3.2.1. Record the room temperature as displayed on the room thermostat.
- 3.2.2. Using the thermometer provided, determine and record the temperature of the water contained in the 1L flask.
- 3.2.3. The square plastic weigh boat will be used for collecting the water. The same weigh boat can be used the measurements of each volume.
- 3.2.4. With the pan empty and all the doors closed, tare the balance to ensure it begins at zero.
- 3.2.5. Place the weight boat onto the pan, close all doors and press tare; 0.000 (or 0.0000) should appear in the display.
- 3.2.6. Begin with a pipette and associated tip for 7 μ L and draw up the appropriate amount of water.
- 3.2.7. Dispense the water from the pipette into the weigh boat that is on the scale pan and close the doors. Allow the display to stabilize and record the weight.
Note: Due to the small volumes and associated weights, the container can remain on the pan and liquid dispensed directly into the container. IF you choose to remove the container when dispensing the liquid, be sure to wear gloves when touching the weigh boat. The oils from your skin can (may) have an effect.
- 3.2.8. Tare the balance and repeat dispensing the pipette volume into the weigh boat, taring between each measurement.
- 3.2.9. Repeat steps 3.2.6 – 3.2.8 for the remaining volumes.

VOLUMETRIC PIPETTE VERIFICATION**General Instructions**

1. Ensure top-loading balance (no doors) is level and pan is clean.
2. Handle weights with gloves on, touching only the knob at the top, or using the forceps provided.
3. Zero (tare) the balance at the start of a measurement.
4. Record the weight, including all decimal points displayed.
5. Sign/date the data with your first initial and last name.

A. Scale Calibration (Verification)

Weight Set Serial Number: _____

Balance ID Number: _____

Balance sensitivity (number of digits to right of decimal): _____

NOTE: tare (zero) the balance when there is no weight on the pan.

Reference Weight	Trial #1 (g)	Trial #2 (g)	Trial #3 (g)	Analyst
0.100 g (100 mg)				
1 g				
10 g				
100 g				

B. Pipette Verification

Room temperature: _____

Water Temperature: _____

Note: Refer to lab procedure regarding taring (zeroing) the balance

Pipette Expected Volume	Trial #1 (g)	Trial #2 (g)	Trial #3 (g)	Analyst
20 mL				
10 mL				
5 mL				
2 mL				
500 μ L **				

** Use a micropipette for this volume

MICROPIPETTE VERIFICATION**General Instructions**

1. Ensure analytical balance is level, pan and inside cavity are clean.
2. Doors shall be closed and remain closed during weighing operation.
3. Handle weights with gloves on, touching only the knob at the top, or using the forceps provided.
4. Zero (tare) the balance at the start of a measurement.
5. Record the weight, including all decimal points displayed.
6. Sign/date the data with your first initial and last name.

A. Scale Calibration (Verification)

Weight Set Serial Number: _____

Balance ID Number: _____

Balance accuracy (number of digits to right of decimal): _____

NOTE: tare (zero) the balance when there is no weight on the pan.

Reference Weight	Trial #1 (g)	Trial #2 (g)	Trial #3 (g)	Analyst
10 g				
1 g				
0.100 g (100 mg)				
0.010 g (10 mg)				
0.005 g (5 mg)				

B. PIPETTE VERIFICATION

Room temperature: _____

Water Temperature: _____

Note: Refer to lab procedure regarding taring (zeroing) the balance

Pipette Expected Volume	Trial #1 (g)	Trial #2 (g)	Trial #3 (g)	Analyst
7 μ L				
50 μ L				
250 μ L				
750 μ L				
2 mL **				

** Use the volumetric pipette for this volume.

Post-Lab Questions

1. Create graph displaying the standard weight results; one graph for the top loading balance and a second for the analytical balance.

Were the results linear? Top loader: yes no Analytical: yes no

Would additional intermediate standards be helpful on either balance? Why? _____

2. Using the density of water from the table on page 2, calculate the volume of water for each pipette and the resulting average.

Pipette (expected volume)	Trial 1 (mL)	Trial 2 (mL)	Trial 3 (mL)	Average (mL)
2 mL				
5 mL				
10 mL				
20 mL				
Bonus 500 μ L				
Pipette (expected volume)	Trial 1 (μ L)	Trial 2 (μ L)	Trial 3 (μ L)	Average (μ L)
7 μ L				
50 μ L				
250 μ L				
750 μ L				
Bonus 2 mL				

3. Reflect on the actions taken and data recorded during the laboratory, considering the questions below. Review the calibration fundamentals covered during lecture and incorporate the concepts in your answers.

How are the differences in the scale sensitivity reflected in the volume results?

Would it be possible to calibrate the 7 μL or 50 μL on the top loader?

Would it be possible to calibrate the 10 or 20 mL on the analytical balance?

What challenges are presented when using the standard weights?
