Laboratory Experiment 1. Calibration of Volumetric Glassware

An important trait of a good analyst is the ability to extract the best possible data from his or her equipment. For this purpose, it is desirable to calibrate your own volumetric glassware (burettes, pipettes, flasks, etc.) to measure the exact volumes delivered or contained. Volumetric glassware can be calibrated by measuring the mass of water they contain or deliver. Glass transfer pipettes and plastic micropipettes can be calibrated by weighing the water delivered from them. A volumetric flask can be calibrated by weighing it empty and then weighing it filled to the mark with distilled water.
This experiment also promotes improved technique in handling volumetric glassware.

Calibrating 25 mL pipette

Correct use of the pipette:

1. Make sure that the pipette filler is dry and that any solution previously used does not contaminate your sample. Fit it to the pipette and check that it is working properly *i.e.* it draws solution into the pipette and the pipette does not leak when held vertically. Do not force the filler onto the pipette in trying to stop a leak - the pipette is likely to break and cause an injury.
2. Rinse the pipette with the solution it is to contain.
3. Fill the pipette again, so that the solution is above the graduation mark and has no bubbles in it.
4. Wipe the outside of the pipette with a tissue or paper towel.
5. Adjust the solution correctly to the mark allowing the bottom of the meniscus to sit upon the graduation mark, with the pipette held vertically and viewed with the eye in line with the mark.
6. Touch the pipette tip against the inside of the vessel from which the solution was taken to remove any drop of solution remaining on the outside of the tip.
7. Release air into the top of the pipette (with some pipette fillers it is necessary to remove the filler from the pipette at this stage) and let the solution drain naturally (by gravity only) into the collecting vessel.
8. Hold the pipette vertically for five seconds after the last drop.

9. Touch the tip against the inside of the vessel - this removes some of the solution held in the tip. The final portion of solution remaining in the tip should not be expelled, because the calibration of the pipette will have allowed for it.
10. Wash the pipette so that solution does not dry in it.

Do not:

a) Blow down the pipette.
b) Hold it by the bulb (hand warmth will alter the volume).
c) Allow solution to dry out in the pipette tip.

Note that you cannot check the accuracy of the pipette unless you know that the balance and the thermometer are accurate too. However, you can estimate the precision with which you can measure 25 cm^3 with the pipette. If the balance is correctly calibrated and set-up properly, the random error due to weighing will be very small compared with that due to pipetting.

Dissolved air in the water affects its density. De-gas the water - the usual techniques are by boiling, using an ultrasonic tank or bubbling helium through it - about 10 minutes for 1 liter.

Inspect the pipette and the pipette filler. Ensure that the pipette is not chipped and that the filler is clean and dry, otherwise the volume measurements will not be correct. Fill the pipette with water and allow it to drain out. Check that the tip is not blocked and the water drains freely. If the pipette is clean no droplets of water will be left on the sides of the pipette. If the pipette is dirty or blocked, clean it or select another one that is clean.

Calibration Procedure.

1. Ask the laboratory supervisor to make the checks normally done in the laboratory to ensure that the balance is working satisfactorily.
2. Check and record the temperature of the water.
3. Make sure that the weighing bottle is clean and dry, put it on the scale pan and tare the balance to zero. An alternative: tare the balance before weighing the bottle, and weigh it.
4. Use the pipette in the recommended manner (see above) to transfer 25 cm^3 of water from the conical flask to the weighing bottle.

5. Return the weighing bottle to the balance and note the weight.

6. Empty the water from the weighing bottle back into the conical flask, dry the beaker with a paper towel.

7. Repeat the steps 3-6 to obtain 10 weighings.

8. Check and record the temperature of the water again.

Calculate the volumes of the 10 pipettings, their mean and standard deviation.

Calibrating a 50-mL Burette

This procedure tells how to construct a graph such as Figure 3-2 in the textbook to convert the measured volume delivered by a burette to the true volume delivered at 20°C.

1. Fill the burette with distilled water and force any air bubbles out the tip. See whether the burette drains without leaving drops on its walls. If drops are left, clean the burette with soap and water or soak it with cleaning solution. Adjust the meniscus to be at 0.00 mL, and touch the burette tip to a beaker to remove the suspended drop of water. Allow the burette to stand for 2-5 min while you weigh a weighing bottle flask fitted with a cup. If the level of the liquid in the burette has changed, tighten the stopcock and repeat the procedure.

2. Drain 10 mL of water slowly into the weighing bottle, and cap it tightly to prevent evaporation. Allow 30 s for the film of liquid on the walls to descend before you read the burette. Estimate all readings to the nearest 0.01 mL. Weigh the weighing bottle again to determine the mass of water delivered.

3. Repeat the procedure for 20, 30, 40, and 50 mL. Then do the entire procedure (10, 20, 30, 40, 50 mL) a second time.

4. Use the table in the textbook to convert the mass of water to the volume delivered. Repeat any set of duplicate burette corrections that do not agree to within 0.05 mL. Prepare a calibration graph like Figure 3-2 in the textbook, showing the correction factor at each 10-mL interval.