USE OF LABORATORY BALANCES AND GLASSWARE

Background Reading: Harris, 7th ed, Chap. 2.; Skoog, West, Holler and Crouch, 7th ed., Chap. 2.

Introduction

The purpose of this exercise is to acquaint you with two of the most commonly used 'Tools of the Analytical Trade': balances and volumetric glassware. The following experiments may appear to be quite simple and straightforward, requiring little attention to detail. NOT SO! Your ability to do well in this course, and more importantly to become a good practitioner of analytical chemistry, requires careful thought and attention. Take these introductory exercises seriously so that you will get in the habit of always doing procedures the correct way.

Experimental

Use of Analytical Balances

Before starting, make sure to read carefully the description of Electronic Balances in Chapter 2. Obtain a weighing sample from the TA. Weigh this sample at least five times on one of the top-loading analytical balances, each time starting the procedure from the beginning (i.e., remove the sample from the weighing chamber, zero the instrument, and then begin anew). BE SURE NOT TO TOUCH THE SAMPLE WEIGHT DIRECTLY WITH YOUR FINGERS. Grease and oils on your hands can add substantially to the weight of the sample. Calculate the average and standard deviation of your replicate measurements. What do you estimate the inherent relative error of your balance to be?

Volumetric Dilutions and Titrations

The goal of this exercise is to sharpen your skills in using volumetric glassware. You will be issued a sample of HCl solution. You will be asked to dilute it and determine the concentration of the diluted solution by titration with a strong base. You will prepare the standard base by dilution of a standard NaOH solution available in the lab.

Exchange a 100-mL volumetric flask from your desk equipment for one of the 100-mL volumetric flasks containing the HCl unknown solution. Record the code number on the table. Dilute the unknown sample to the mark with deionized water. Pipet a 10 mL aliquot of this diluted solution into an Erlenmeyer flask and titrate it with the 0.1 *M* standard NaOH solution you have prepared by dilution of the 1 *M* NaOH supplied. Use phenolphthalein as the indicator. (The exact concentration of the NaOH solution is given on the label.) Calculate the molarity of the HCl solution.

The correct technique for use of a volumetric pipet is shown in the figure below.

Repeat the titration several times to determine the uncertainty in your measurement.

Calibration of a Manual Buret

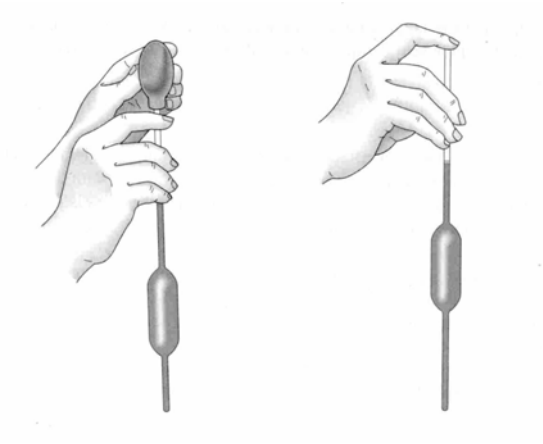
There will be two burets on display in the laboratory; one filled with a clear solution, the other with a colored solution. Read the volume levels on both burets and record the values in your notebook. Before proceeding to the second part of the exercise, show the results to one of the TA's and describe briefly how you read the buret. When the TA has determined that you are reading the burets correctly you may then continue with buret calibration.

Fill a 50 mL buret with distilled water. Force any air bubbles out of the tip. Make sure that the buret drains without leaving any drops on the walls. If drops are left, clean the buret with soap and water. Adjust the meniscus to be slightly below 0.00 mL and touch the buret tip to the side of a beaker to remove the suspended drop. Allow the buret to stand for 5 min while you weigh a 100 mL beaker covered with parafilm. If the level of the liquid in the buret has changed, tighten the stopcock and repeat the procedure.

The proper technique for use of a buret is shown in the drawing below.

Drain approximately 10 mL of water into the weighed beaker and cap it to prevent any evaporation. Allow ~ 30 s for the film of liquid on the buret walls to descend before you read the buret. Estimate buret readings to the nearest 0.01 mL. Weigh the beaker again to determine the mass of water delivered.

Now drain the buret from 10 to about 20 mL and record both the volume and the mass of water delivered. Repeat for 30, 40 and 50 mL of water. Then, repeat the entire procedure two more times.



Use Table 2-3 in Skoog et al. (p 51) or Table 2-7 (p. 32) in Harris to convert the mass of water delivered to a volume. Subtract the apparent volume (the buret reading) from the true volume (calculated from the mass of water delivered). This difference is the correction that should be applied.

Familiarization with an Automatic Buret

You will use the automatic buret for several titrations during the semester. In each case the teaching specialist will be responsible for filling the reservoir, but you will need to learn how to fill the buret from the reservoir, dispense the titrant, and read the volume delivered.

Examine the front panel controls and displays. Note the three control keys— **Fill**, **Clear**, **Go**— and a knob to set the dispense/fill rate. Note also that there is a cable-connected pushbutton switch. This has the same function as the **Go** switch on the panel but provides the convenience of remote handheld operation. The display indicates the volume dispensed and the current operating mode, with DOS ↑ indicating the dispensing mode, in which titrant will be delivered, and DOS ↓ indicating the fill mode, in which the buret will be refilled. The arrow indicates the direction of motion of the piston. When the piston moves up liquid is driven through the delivery tube. When the piston moves down solution is drawn from the reservoir bottle into the buret.

Look at the position of the piston. Is the buret full? If not, press **Fill**. When the buret is full, it will automatically switch back into delivery mode.

If you are beginning a titration you want to reset the volume dispensed to 0.0 mL. Depress the **Clear** key. The display should now read 0.0 mL.

When you are ready to begin delivering titrant, press and hold the **Go** key (or the remote pushbutton). The display should now read DOS ↑ followed by the volume of titrant delivered. The buret will continue to dispense titrant until the **Go** key is released.

You can control the rate at which titrant is delivered (and the fill rate) by adjusting the rate knob. Position 1 is the lowest rate of delivery, and position 10 is the highest rate of delivery.

Report

Report the average, standard deviation, and 95% confidence limits of your weight measurements. What is the inherent ***relative uncertainty*** of the analytical balance you used?

Report the average and standard deviation of the molarity of your HCl solution.

Using Excel, prepare a calibration graph showing the correction factor for your manual buret at each 10 mL interval. Include the experimental uncertainty in your measurements on your graph, here defined as a range of $\pm$ one standard deviation about the mean. Paste this plot into the Report.