Introductory Laboratory Techniques

Introduction

Welcome to the Chemistry 2A Laboratory. You will find that experimentation will help you understand the lecture material since chemistry *is* an experimental science. In the laboratory you will go over many practical applications of the theories you learn in class. Use the laboratory as a study aid to help you understand chemistry and perhaps you will even have some fun!

Many students do not enjoy laboratory and do not find it helpful because they take a "cookbook" approach to chemistry. That is, they are thinking, "I mix 1 gram of this with 5 mL of that to get a blue solution with white stuff at the bottom". They do nothing more than follow the recipe without thinking about what is happening in the test tube and how it relates to what they are studying and to the rest of the world in general. Since we don't let you eat the end results of what you cook in lab, if you take the cookbook approach you are going to have a poor experience in the laboratory and an especially hard time completing your laboratory reports.

This lab manual is written to help you avoid such a bad experience and to help you develop skills in solving problems. You will not find recipes in your experiments; you are given considerable leeway in designing your own experiments. Whenever you need a lab technique you will be given complete instructions on how to execute it, but you must be able to figure out how to apply those techniques in discovering the solutions to the problems presented. It is critical that you read the experiment *before* coming to the laboratory, and attempt to understand the theory behind the experiment and the methods you will use in the laboratory to investigate that theory.

Consider yourself an *investigator* while you are in the laboratory. For example, in a typical reaction first find out "who done it"; what chemicals take part in the reaction? Then find out the culprits' "method"; is energy taken in or given off? Finally you need to find out the consequences; what compound is formed? If you take this approach you will have a better laboratory experience, and you will have a much easier time writing the experimental report. In short, you will learn more and learn more easily.

This lab is designed to 1) acquaint you with the equipment in your locker, and 2) introduce you to some basic laboratory techniques. A word of warning: a few of you may find this and other beginning laboratories in Chemistry 2A to be somewhat tedious, especially if you've had a good high school chemistry laboratory course. However, please be patient as the goal is to give all students a good common background so every student has an excellent chance of success with the later, more difficult experiments. Remember that as pre-laboratory preparation, you should come to the laboratory with Title, Purpose, Procedure, and Data Tables entered in your notebook. At the end of the laboratory period you should have your TA sign and date your laboratory notebook near your data tables. At the next laboratory meeting you will submit a completed report.

Common Laboratory Procedures

You will now do a set of simple experiments to teach you the proper techniques for using the different equipment in the laboratory. You must read the common laboratory procedures section of this manual before beginning this part of the exercise. These pages describe the proper use of equipment.

In these procedures you will learn to properly:
a) use a balance,
b) measure the volume of a liquid, and
c) use a Bunsen burner.

A record of all data should be placed in your laboratory notebook. All calculations should be carefully and clearly shown as well. Finally, be sure to answer all questions before turning in your report to the teaching assistant.

Safety: Wear your goggles. Use a bulb with an Eppendorf tip to fill a pipet.

Procedure

Work individually on experiment.

Part I. Measuring Volumes

A. Using a Pipet to Measure Volume

1. Draw about 400 mL of deionized water into a clean beaker, and let it stand for 15 minutes to equilibrate to room temperature. Note, there is only one deionized water tap in the lab room; make sure you use the correct tap.

   Helpful hint: You will need to take turns with your locker-mate using the 10.00 mL volumetric pipet. One of you should start part B, using a buret to measure volume, while the other is doing part A.

2. Confirm that your 10.00 mL volumetric pipet is clean by filling to above the mark with deionized water and then letting it drain. Your pipet is a transfer pipet that is calibrated “to deliver” (TD) rather than “to contain” (TC). The last drop of liquid should not drain out of the tip of a TD pipet in normal use. However, there should be no water drops left on the side walls. The presence of such drops indicates that your pipet is dirty.

   Helpful hint: You will use your pipet in many of the experiments in Chemistry 2. **It is important that you always clean it at the end of the day, and rinse it thoroughly with deionized water before storage**. Pipet cleaning solution is located in a 1 L bottle at the front of the room. Follow the instructions on the label. Remember that your pipet is calibrated to deliver.

3. Measure and record the mass of a clean and dry 50 or 125 mL Erlenmeyer flask.

4. Measure and record the temperature of the room and of the water that was set aside in step 1. The two temperatures should agree before you continue. Read the thermometer to the closest one tenth of a degree, using your best estimate. Please be especially careful with the thermometer.

5. Use your pipet to deliver 10.00 mL of the equilibrated water into the Erlenmeyer flask. Note the precision used here.

6. Measure and record the mass of the flask and the water.

7. Repeat steps 5 and 6 at least two additional times without emptying out your flask between trials.

8. Calculate the mass of water delivered by your pipet for each trial. Use your mass of water and the volume of the volumetric pipet to calculate the density of water for each trial. Calculate the average density, standard deviation, and the 90% confidence limits for the average density.

9. Use the temperature of your water along with the values of mass and volume of water given in Table I to calculate the accepted values for the density of water.

10. Determine the relative error with respect to the average density of water. The relative error is defined by:

$$\text{relative error} = \frac{|\text{experimental result} - \text{accepted value}|}{\text{accepted value}} \times 100$$

Table I. The volume occupied by 1.0000 g water weighed in air against stainless steel weights.

Temperature (°C)	**Volume (mL)**
18	1.0024
19	1.0026
20	1.0028
21	1.0030
22	1.0033
23	1.0035
24	1.0037
25	1.0040
26	1.0043

Table I gives the corrected volume in mL occupied by 1.0000 g of water when weighed in air against stainless steel weights for different temperatures. Two effects are included in this volume per 1.0000 g; first, the change in the density of water with temperature; and second, a much smaller correction due to buoyancy. The buoyancy correction arises since the balance was set to zero with a certain mass of air on the balance pan. The volume of water displaces some of this air from the balance pan that makes the water appear lighter than it really is. The contribution of buoyancy to the results in Table I is roughly 0.0011 mL per 1.0000 g of water.

B. Using a Buret to Measure Volume

1. Discard the water in your Erlenmeyer flask, and remeasure the mass of the flask. The inside of the flask need not be completely dry because any water left in it is from the previous procedure and is at the same temperature as the new water you will be adding.

2. Use a 25 mL buret and **accurately** measure out *about* 24 mL of room temperature deionized water from part A into the flask. You should read the buret to the closest one hundredth mL (e.g., 24.14 mL). In your laboratory notebook, record your initial buret reading and your final buret reading. The volume of water delivered by the buret is the difference between the final and initial buret reading.

3. Measure and record the mass of the flask and the water.

4. Repeat steps 2 and 3 at least two additional times without emptying out your flask between trials.

5. Calculate the mass of water delivered by your buret for each trial. Use your mass of water and the volume of the water delivered by your buret to calculate the density of water for each trial. Calculate the average density, standard deviation, and the 90% confidence limits for the average density.

6. Assuming that the water temperature has not changed, compare your experimental value of the density of water to the accepted value of the density of water you calculated in part A.

7. Determine the relative error with respect to the average density of water when measured by the buret.

Always clean your buret after use and rinse it with deionized water before storage. Furthermore, be sure you follow the instructions given at the beginning of this manual for proper use of the buret.

C. Using a Flask to Measure Volume

1. Measure and record the mass of ANOTHER CLEAN and DRY 50 or 125 mL Erlenmeyer flask. Note this flask needs to have a 50 mL graduation mark.

2. Use your CLEAN and DRY 50 or 125 mL Flask and carefully measure out 50 mL of your room temperature water.

3. Measure and record the mass of the flask and the water.

4. Empty out your flask and carefully measure out another 50 mL of your room temperature water. There is no need to reweigh the empty flask.

5. Measure and record the mass of the flask and water.

6. Repeat steps 4 and 5 at least once more

7. Use your mass of water and the volume of the water measured by your Erlenmeyer flask to calculate the density of water for each trial. Calculate the average density, standard deviation, and the 90% confidence limits for the average density.

8. Assuming that the water temperature has not changed, compare your experimental value of the density of water to the accepted value of the density of water you calculated in part A.

9. Determine the relative error with respect to the average density of water when measured by the flask.

Part II. Drying a Hydrate

1. As illustrated by the TA, place a clean crucible on a wire triangle above a Bunsen burner. With the TA watching, light the Bunsen burner and adjust the flame and the height of the wire triangle so that the crucible is positioned in the hottest part of the flame.

2. Heat the crucible for five minutes to make sure it is dry, and then remove it from the wire triangle using crucible tongs and place it on your desktop to cool.

3. After the crucible has returned to room temperature (approximately five minutes), measure and record its mass.

4. Weigh into your crucible 1.0 - 1.2 g of manganese(II) sulfate monohydrate, $MnSO_4 \bullet H_2O$.

5. Heat the crucible with its contents for five minutes, and then remove it to your desktop using crucible tongs.

6. After the crucible and its contents have returned to room temperature, measure and record the mass.

7. Repeat steps 5 and 6 until the mass readings are consistent. (Mass no longer decreases after heating.)

8. Calculate the mass loss by your sample upon heating.

9. Transfer the contents of your crucible to the waste container located in the fumehood.

Clean-up: **Solid dry manganese(II) sulfate may be disposed of in the proper waste container found in the fumehood**. Clean your volumetric pipet and buret with deionized water only. All other glassware may be cleaned with tap water and rinsed with deionized water. Always, let your glassware air-dry. If time permits, now would be a good time to also clean any other dirty glassware in your locker. Be sure that all glassware is returned to the proper place, and that your laboratory bench has been rinsed with water using a sponge. Inform your TA that you are leaving and be sure that your laboratory drawer gets locked.

Write-up: Data Tables should contain the measured volumes and masses of trials in Part I and all masses measured in part II. Be sure to include units on your measurements. Show calculations for at least one trial. Show all calculations for averages, standard deviations, 90% confidence limits, and accepted values for density.

The Results Section of the laboratory write-up should include tables of any calculated values in this laboratory. Also, discuss how the relative error with respect to the average density of water when measured by the pipet, buret, and Erlenmeyer flask differ. Indicate the relative accuracy of each of the pieces of glassware you have used to measure volume. For part II discuss why the sample lost mass.

Questions

Part I

Question A: If you wanted to accurately measure a 20.00 ± 0.02 mL volume of a liquid, which piece of glassware would you use; 10.00 mL pipet, buret, or Erlenmeyer Flask? If you wanted to quickly measure about 20 mL, which would you use?

Question B: Generally, a pipet is more accurate than a buret. Do your experimental results support this statement? If yes, explain. Explain why a student's experimental results may not support the statement?

Part II

Question C: What is the chemical formula of the final product?

Question D: What is the theoretical percent mass of water in manganese(II) sulfate monohydrate? Calculate your experimentally determined percent mass of water in manganese(II) sulfate monohydrate. What is your relative error with respect to percent mass of water in the monohydrated sample?

Question E: Why did your empty crucible need to be dried by heating? If your crucible had not been dried before heating the sample how would that have affected your calculations in Question D?