

Introductory Lab for General Chemistry

(Adapted from *Microscale General Chemistry Laboratory with Selected Macroscale Experiments*, Second Edition by Zvi Szafran, Ronald M. Pike and Judith C. Foster, Wiley, 2003)

Objectives Assign lockers for each pair of students and describe aspects of the course syllabus that pertain to lab attendance, lab reports and schedule of experiments.

Provide general safety rules designed to protect all laboratory workers and their working environment. Review preventative as well as emergency safety measures to be followed throughout this course. This includes attire, as well as handling of chemicals and equipment. Note location of all safety equipment in the lab.

Review proper expression of chemical measurements including scientific notation, significant figures and graphing.

Become familiar with laboratory equipment, and experimental techniques such as transferring solid and liquid chemicals, weighing, heating, stirring, filtering, preparing solutions and titrating. Note items found in each student drawer and locker and inventory these items.

Perform select sections of Experiment 1: Basic Laboratory Measurements designed to teach the laboratory worker proper use of balances, graduated cylinders, pipets, and filtration techniques.

Succeed at using correct significant figures in recording measurements taken as well as in calculating and reporting answers.

Required Ch 1-3 and Exp. 1 pages 1-61 in Microscale General Chemistry Laboratory
Prior Reading 2nd Ed. by Szafran, Pike and Foster

Section 1.5 and Appendix 1 in Chemistry, 3rd Ed. by Julia Burdge

Introduction A successful learning experience in general chemistry must be accompanied by correlated laboratory investigations that relate the topics learned in the classroom to tangible applications. These investigations must be performed safely, using correct techniques and results must be reported properly. This introductory lab is designed to insure these outcomes for the rest of the course and the lab worker's scientific career.

Procedure Part A

Read the College of New Jersey Department of Chemistry Safety Rules provided. Pay special attention to the details pertaining to proper dress. **YOU MUST WEAR eye protection, closed toed shoes, and long sleeves and long pants.** Your clothes, especially “tops” (shirts, sweatshirts, etc) should be close-fitting and non-synthetic at all times. Complete the form acknowledging familiarity and intent to comply with these rules and turn it in to your instructor.

Discuss the benefits of using the microscale technique in this course such as generating less toxic waste, reducing the chance of fire, improving lab air quality and minimizing exposure to dangerous chemicals. Review proper handling of chemicals and equipment especially never work alone in the lab, never directly smell or taste a chemical, never return sampled reagent to a container of stock solution, immediately recap containers and don't contaminate the cap while it is off the container, never heat a closed system, keep your workspace clean and uncluttered, don't add water to dilute acids, and dispose of waste only in the proper containers designated for it. Note the location of the following in your lab and draw a diagram of the room including: the safety shower, the fire extinguishers, the fire alarm, all exits, the fire blanket, the eyewash station, the first aid kit, all the fume hoods, the broken glass container, the safety goggles drawer and your lab drawer location. Download and print a Material Safety Data Sheet and highlight the following items on it: the CAS#, fire and explosion data, and protective equipment required when working with that chemical.

Your instructor will review scientific notation and performing mathematical operations such as addition, subtraction, multiplication and division in scientific notation. Your instructor will review significant figures. You will learn that all the digits given by the electronic balance are significant and should be written on your data sheet. Other measurements such as those taken from the barometer, a thermometer, a ruler, a graduated pipet, buret or a graduated cylinder, are significant to one place beyond the digit for which the device has hash marks (or graduations). For example, a thermometer with lines for every degree, can be read with the tenths of a degree estimated so all temperatures taken with it should be reported with one decimal place (even if that decimal place is a zero).

Writing your measurements with the correct number of significant figures is only the first part of this indicator of uncertainty. Learning which digits are significant in a number, and how to perform operations such as addition, subtraction, multiplication, division and taking a log with correct significant figures reported in the answer will also be taught.

All non-zero digits are significant.
Zeroes between non-zero digits and zeroes to the right of a number that follows a decimal point are significant.
Zeroes to the left of a non-zero digit are place-holders and thus not significant. Writing the number in scientific notation can show this fact.
Zeroes to the right of a non-zero number are not significant. To make them so, write them in scientific notation or place a period after them.
Exact numbers and conversion factors that are a definition are all significant.

In addition and subtraction, your answer can only have as many decimal places as the least number of decimal places in the numbers being added or subtracted. Don't look at how many significant figures that is until after you have completed the operation.

In multiplication and division your answer can only have as many significant figures as the least number of significant figures in the factors being multiplied or divided.

When taking the log of a number your answer will have as many decimal places as the number of significant figures in the number whose log you are taking.

Keep track of how many significant figures each step of a multi-step problem allows, but don't round off at each step. Keep an extra significant figure at each step, and round off to the correct number of significant figures at the end. When rounding to a particular number of significant figures, round up if the digit being dropped is >5 and stay put if the digit being dropped is <5 . If the digit being dropped is exactly 5, round up if the preceding digit is odd and stay put if the preceding digit is even.

Note the glassware and equipment found in your student drawer and locker. Make sure you are familiar with the name of each item and indicate the presence or absence of each item listed on the inventory sheet you will be provided. If necessary, go to the stockroom and obtain items missing from your drawer or cabinet.

Your instructor will review proper experimental techniques such as using a spatula for only one reagent to avoid cross-contamination, keep pipets vertical at all times, immerse only their tip in a reagent solution, never lay stoppers on a bench, pour liquids down a glass rod to avoid spills, never weigh directly on a balance pan (always use a weighing paper or weighing boat) never use a beaker or Erlenmeyer flask to measure a volume, always clamp vessels to a ring stand when filtering, note that some graduated pipets deliver their volume when all is allowed to flow out while others must be stopped at the marked line in order to deliver their volume, always read a meniscus at eye level, etc.

Part B & C

Basic Laboratory Measurements

Compare the accuracy of volume measurements taken with a graduated cylinder, a graduated pipet and an automatic delivery pipet.

B1 Place about 50 mL of tap water in a beaker (thereafter the “water supply beaker”), allow it to equilibrate to the lab temperature and measure its temperature. Note the hash marks on the thermometer, estimate your temperature to the next decimal place from that for which there are hash marks, and write the temperature on your data sheet with the correct number of decimal places.

Measure 2.0 mL of water from the “water supply beaker” whose water temperature you know in a 10-mL graduated cylinder, reading the volume from the bottom of the meniscus while holding the cylinder at eye level. Note the hash marks on the cylinder, estimate your volume to the next decimal place from that for which there are hash marks, and write the volume on your data sheet with the correct number of significant figures.

Transfer ALL of the water in your cylinder into a tared 10-mL Erlenmeyer flask and write the mass of your water with all the digits given by the balance on your data sheet.

Use a table of densities of water at various temperature to calculate the volume of water actually transferred using $\rho = m/V$.

B2 Use a 2 mL graduated pipet and a pipet pump to draw water from the “water supply beaker” whose temperature you know. Draw water to a little bit above the 0 mark (but don’t draw water into the pump), remove the pump and replace it with your DRY forefinger. Allow the fluid to flow down to the meniscus by adjusting the pressure applied with your forefinger. Wipe any excess water off the outside of the pipet, and allow 2.0 mL of water to flow into a tared 10-mL Erlenmeyer flask. Note which type of pipet you are using to make sure you allow the water level to drop completely out of the pipet or if you must stop it after dispensing 2 mL by applying pressure with your finger.

Write the mass of your water with all the digits given by the balance on your data sheet. Use a table of densities of water at various temperature to calculate the volume of water actually transferred using $\rho = m/V$.

B3 Use an automatic delivery pipet whose volume is dialed to 500 μL to measure 500 μL of water from the “water supply beaker” whose temperature you know. To do so, depress the plunger of the pipet to the 1st stop, insert the pipet tip into the water, draw up the designated volume of water, lift the pipet

out of the water and place it over the mouth of a tared 10-mL Erlenmeyer flask, and depress the plunger to the 2nd stop releasing the entire contents of the tip. Write the mass of your water with all the digits given by the balance on your data sheet. Use a table of densities of water at various temperature to calculate the volume of water actually transferred using $\rho = m/V$.

Calculate the % error of the volume dispensed by each device using

$$\% \text{ error} = (\text{measured volume} - \text{calculated volume}) / \text{calculated volume} \times 100\%$$

Determine the solubility of KNO_3 in water at 0°C

C1 Weigh 1.5 g of KNO_3 on a tared piece of glazed weighing paper and write the mass of your salt with all the digits given by your balance on your data sheet. Transfer the KNO_3 to a clean 25-mL Erlenmeyer flask. Measure 5.0 mL of water with a graduated cylinder and add it to the salt-containing flask. Record the volume of water added, with the correct number of significant figures, on your data sheet.

C2 Place your flask on a hot plate and stirrer with a magnetic stir bar to heat your solution until all of the salt has dissolved. Use a test tube holder to grasp the neck of the Erlenmeyer flask and place it in a water bath to cool. Once at room temperature, place the flask in an ice bath and allow it to cool for 10 minutes.

C3 While the solution cools, set up a suction filtration using your Hirsch funnel, filter flask and adapter. Place a small filter paper in the funnel. Secure the filtration unit to a ring stand and add a couple of drops of deionized water to the filter paper to moisten it.

C4 Record the temperature of the cold solution, turn on the suction and quickly swirl the flask to loosen the precipitated solid and transfer the liquid and solid to the funnel. Insure a quantitative transfer by scraping all the solid with a rubber policeman but do not use water to rinse the solid onto the filter paper (you would dissolve some of your precipitate by doing so). Apply suction for 3-4 minutes to suction-dry the solid.

C5 Weigh the solid KNO_3 on a tared piece of glazed weighing paper and enter the mass of the precipitated salt with all the digits given by your balance on your data sheet.

C6 Calculate the solubility of KNO_3 in cold water using
$$\text{Solubility} = (\text{Original mass } \text{KNO}_3 - \text{Precipitated mass } \text{KNO}_3) / \text{Volume of water}$$

Pre-Laboratory Questions:

Refer to Ch 1-3 in lab manual for reference

Name _____

Date _____

1. List 3 dangers of wearing open-toed shoes in the laboratory.
2. List 3 problems that can stem from leaving spilled chemicals around the balance.
3. List 3 items of clothing or protective wear that you must wear in the laboratory.
4. Write the following numbers in scientific notation and tell how many significant figures are in each.

0.00320	2.5020
21,500	21,500.
507	507.02
507.020	690
5. A ruler has 10 lines between the marks for 1 and 2 cm. You are measuring a line that starts at 0 and ends at 5 on this ruler. What is the measurement with correct significant figures _____cm
6. What is the density of an object with a mass of 15.894g and a volume of 5.05mL? Report your answer with the correct number of significant figures.
7. What is the result of $(12.01 - 11.6) \times 22 =$ _____ with correct significant figures?

Data and Results:Name _____
Date _____**Part B Accuracy of Volume Measurements****B1. Graduated Cylinder**

Temperature of Water	_____ °C
Volume of Water measured	_____ mL
Mass of Water	_____ g
Volume of Water calculated	_____ mL
Density of water at that temperature	_____ g/mL
% error	_____ %

B2. Graduated Pipet

Temperature of Water	_____ °C
Volume of Water measured	_____ mL
Mass of Water	_____ g
Volume of Water calculated	_____ mL
Density of water at that temperature	_____ g/mL
% error	_____ %

B3. Automatic Delivery Pipet

Temperature of Water	_____ °C
Volume of Water measured	_____ mL
Mass of Water	_____ g
Volume of Water calculated	_____ mL
Density of water at that temperature	_____ g/mL
% error	_____ %

Part C Solubility of KNO₃ in Ice Water

Original Mass of KNO ₃	_____ g
Volume of Water	_____ mL
Temperature of the Cold Solution	_____ °C
Precipitated KNO ₃ Mass	_____ g
Mass of KNO ₃ Dissolved	_____ g
Solubility of KNO ₃ in Ice Water	_____ g/mL

Remember to attach your lab diagram and MSD sheet with the required info on each.

Post-laboratory Questions:

Name_____

Date_____

1. Draw a Hirsch funnel (draw both a side and top view) and list another name for it.

2. What is the best piece of glassware in your drawer with which to measure 20 mL of water?

3. Based on your results, which volume measuring device was the most accurate and why?

4. Your partner tells you that she measured two mL of water using your 10-mL graduated cylinder and that volume of water weighed two grams on the balance in our lab. What is the density of the water at the temperature at which she took the measurements? Show the calculation and answer with units and significant figures.