Experiment 1D

FV 8/23/04

INTRODUCTION TO THE CHEMISTRY LABORATORY

PRELIMINARIES:

Prior to doing any independent laboratory work, students must be oriented to several important issues, including (1) plebe chemistry laboratory learning objectives, (2) laboratory safety, (3) laboratory policies of their particular instructor, and (4) basic laboratory procedures and commonly used resources.

Your instructor will address each of these issues at the beginning of the laboratory period. The *general* plebe chemistry laboratory learning objectives are summarized on the last page of this handout; you may want to save this page in your study materials in a prominent location. Each individual experiment will also include a set of specific learning objectives. You will be required to sign and turn in the "Safety Agreement" which is the next-to-last page of this handout following the safety instruction. Take note of your instructor's laboratory policies, including lab partner arrangements, lab report formats, collaboration guidelines, use of the "MOLS" system, etc. These policies may be part of your instructor's Course Policy Statement.

Following the instruction on basic laboratory procedures and resources, you will be expected to know, among other things, the following:

- How to correctly determine masses using both top-loading and analytical balances
- Where and how to obtain solutions for use in experiments
- Where and how to obtain needed glassware and other equipment for use in experiments
- Where and how to dispose of chemicals or mixtures used in experiments

MATERIALS: 50-mL buret, small beakers, 25-mL pipet, 25-mL graduated cylinder, 25-mL volumetric flask, plastic pipet, detergent solution for cleaning glassware, unknown liquid/solid samples, capped plastic weighing vials

PURPOSE: The purposes of this experiment are to become familiar with the common types of laboratory glassware and their appropriate uses; to determine the density of water to a high degree of precision; and to determine the density of an unknown solid or liquid.

LEARNING OBJECTIVES: By the end of this experiment, the student should be able to demonstrate the following proficiencies:

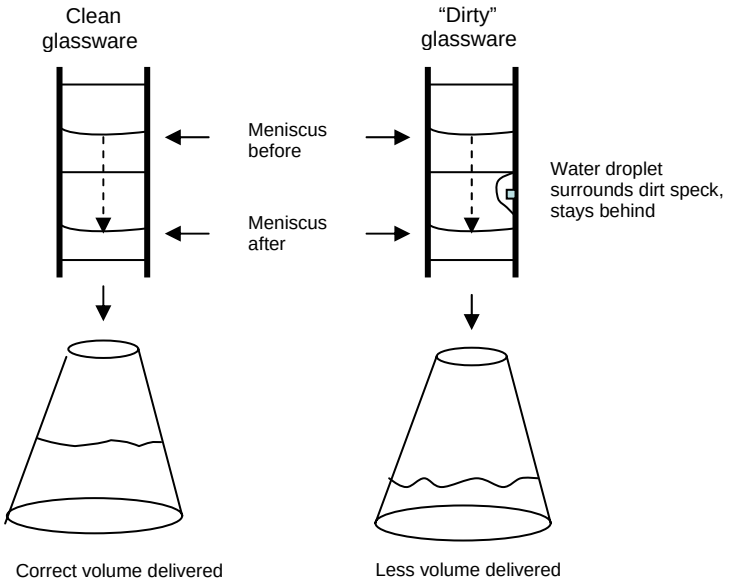
1. Use Excel to graph data and determine the best-fit straight line
2. Understand how multiple data points increase precision
3. Know which glassware should be used in various circumstances
4. Know how to correctly use burets, pipets, and volumetric flasks
5. Devise a simple laboratory procedure for determining the density of water and of an unknown material

6. Determine the average and standard deviations of sets of measurements
7. Recognize and remove bad data points
8. Calculate density to the correct number of significant figures, based on experimental measurements

DISCUSSION:

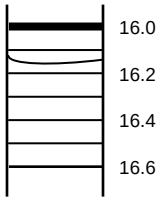
Laboratory glassware. There are two major categories of laboratory glassware: (1) those that contain a certain volume (volumetric flasks) and (2) those that deliver a certain volume (pipets, burets, and graduated cylinders). Some glassware is very carefully designed and marked for high accuracy/precision work (burets, pipets, and volumetric flasks), while other glassware is not intended for such work (beakers, flasks, and graduated cylinders).

In high accuracy/precision work, the glassware must be clean. Not only does clean glassware avoid unwanted chemical contamination, but it also assures that delivered volumes of liquids will be correct. A dirty spot on the inside wall of a buret or pipet, for example, even if the spot itself does not occupy a significant volume, can cause a droplet of water to adhere to the wall, causing an error in the recorded volume of delivered liquid. As illustrated below, only clean glassware will, in general, deliver the volume of liquid corresponding to the recorded volume. If the glassware is dirty, and during delivery the liquid level falls below a dirt speck to which a droplet of water adheres, the same *recorded* volume of delivered liquid does not, in fact, correspond to the actual volume delivered.



(Recorded volume same in both cases)

Measuring and recording data. The generally accepted rule for measuring volumes is to estimate one more digit beyond the digit associated with the closest spaced markings.



Correct reading: 16.16 mL

For measurements obtained from devices which provide digital output, such as the electronic mass balances, the digits should all be written, including any trailing zeroes, with the understanding that the last digit is within "one" unit of the correct value.

Appendix E gives more detailed information about equipment precision, including the various types of glassware to be investigated in this experiment.

Error Analysis. Appendix J of the plebe chemistry laboratory manual is a basic introduction to the kinds of measurements most often encountered in the chemistry laboratory, along with methods for assessing the reliability of these measurements. Please study this appendix carefully. Your instructor may provide additional information as well.

PROCEDURE:

[NOTE: As needed, you will need to organize the way in which you collect and record data. No pre-formatted data sheets are provided.]

1. Observe the correct usages of each type of laboratory glassware (buret, volumetric pipet, volumetric flask, graduated cylinder, beaker, and plastic pipet), as demonstrated by your instructor. Make any special notes for future reference.

2. Devise a procedure for and determine the density of water (to four significant figures) at room temperature, using a buret, a mass balance, and a spreadsheet program.

3. With the calculated density of water, devise a procedure for and determine the volumes of water either contained in or delivered by the common pieces of laboratory glassware available (i.e., 25-mL volumetric flask, 25-mL graduated cylinder, small beaker, 25-mL pipet). Make sure that your procedure results in an average volume contained/delivered (hence, a measure of the accuracy of the glassware) as well as a measure of the average deviation of measurements (hence, a measure of the precision of the glassware).

4. Using the calculated density of water, determine the average volume of water droplets delivered by a plastic pipet, as well as the average and standard deviation.

5. Using the calculated density of water, determine the density of an unknown material to at least three significant figures using the 25-mL volumetric flask, a mass balance, and water. Unknowns will be assigned by the instructor. [SAFETY NOTE: Any materials containing hydrocarbon liquids must be disposed of in the labeled container in the fume hood.]

QUESTIONS FOR CONSIDERATION:

(Follow the guidance provided by your instructor regarding these questions.)

1. In the determination of the density of water, using the buret, what would be the effect on the calculated density if the buret had been dirty, resulting in droplets of water adhering to the inside of the buret? Justify your answer.

2. The density of an unknown liquid is to be determined using a 25.00 mL pipet. Based on the data below, determine the density.

Mass of empty receiving flask:	36.78 g
Mass of flask and unknown liquid after adding from pipet:	54.88 g

   Would using the higher precision mass balance make for a better result here? Why or why not?

   Explain why a more accurate result would probably be obtained if the density was based on more than one trial.

3. What conclusions can you draw from volumes delivered by graduated cylinders or beakers? Would the same conclusion apply to the volume contained in an Erlenmeyer flask? Explain.

4. Suppose that a step in a laboratory procedure calls for making 100.0 mL of solution. Which piece of glassware would be best for this procedure?

   Suppose a procedure calls for adding about 10 mL of an acid solution to a reaction mixture in a beaker. Which piece of glassware would be best for this procedure?

5. In the determination of the volume of droplets delivered by a plastic pipet, why is the standard deviation of the set of measurements larger than the average deviation?

   What can you say about using a plastic pipet as a *precise* volume delivery system?

   Were any of the droplet measurements worthy of omission from the data set? How might the data for such "droplets" have arisen?

6. Based on the data below, determine the density of an unknown ionic solid, using a 50.00 mL volumetric flask, the unknown liquid in #2 above, and a top-loading balance. Assume that the solid is completely insoluble in the unknown liquid.

Mass of empty volumetric flask:	88.43 g
Mass of flask plus added solid sample:	96.21 g
Mass of flask and contents after filling to mark with unknown liquid:	130.02 g

   What can be said about this density determination if it is later noticed that there are a couple droplets of liquid adhering to the neck of the flask *above* the mark?

CHEMISTRY LABORATORY SAFETY AGREEMENT

Before working in the chemistry laboratory, read carefully the safety precautions and techniques for handling chemicals described in http://chem.mathsci.usna.edu/manual/safety.pdf. Give this agreement, signed and dated, to your laboratory instructor on the first day of lab.

When you are in the laboratory, THINK about what you are doing at all times.

1. Wear approved chemical splash goggles in the laboratory. If any hood sash is up, you must wear goggles.

2. Do not attempt any unauthorized experiments.

3. Know the location and operation of safety equipment.

4. Bring only necessary materials into lab. Book bags, jackets, etc., are to be left in the hallway.

5. Never work alone in the laboratory.

6. Never eat or drink in the laboratory.

7. Use the fume hood when necessary.

8. Keep your work area uncluttered.

9. Use only equipment that is in good condition.

10. Dispose of waste and excess materials according to your laboratory instructor's directions.

11. No horseplay in the laboratory.

12. Don't sit or lean on laboratory work surfaces.

13. Handle chemicals with caution.
    (a) Read labels carefully.
    (b) Use only the amount required.
    (c) Leave reagent containers in their proper places.
    (d) Clean up all spills immediately.
    (e) Label all chemical containers.

14. Thoroughly wash your hands any time you leave the laboratory.

15. Immediately report all accidents and physical/chemical injuries, no matter how minor, to your laboratory instructor. Be ready to take immediate action as needed to assist any injured classmate.

16. Don't leave the laboratory without your instructor's approval.

I have carefully read all the safety precautions on the pages at the website above and recognize that it is my responsibility to observe them throughout my chemistry course.

Name _____ Signature _____

Course _____ Section _____

Instructor _____ Date _____

Plebe Chemistry Laboratory Learning Objectives **(6 Aug 2004)** **Plebes should be able to:**
Understand and apply proper safety measures in the laboratory
Perform data analysis with Excel (basic spreadsheet calculations, graphing, regression, use of functions)
Understand and apply accuracy and precision in measurement and calculations (correct significant figures in measurement; correct sig figs in calculations involving measurements)
Perform error analysis (simple averaging, simple and standard deviation, quality of straight line fitting)
Understand and apply straight line equation modeling
Prepare solutions, perform titrations, mass measurements, appropriately use glassware, and perform other simple laboratory procedures
Understand and apply the concept of calibration
Use chemical instrumentation (Spec-20; pH meter; electrometer)
Perform pressure measurements (proper accounting for specific conditions of measurement)
Organize data gathering (e.g., preparing data sheets)
Write coherent laboratory reports in appropriate formats, including drawing correct conclusions from analysis of experimental data
Achieve quality results (both quantitative and qualitative)
Design some laboratory procedures (with guidance)
Demonstrate learning through assessment (via tests, lab quizzes, lab practicals, etc.)
Demonstrate integration of concepts of laboratory with those associated with other aspects of general chemistry course and with important real-world applications, including naval applications
Work appropriately with other midshipmen in various kinds of collaborative arrangements, both in and out of the laboratory setting