LB 171L: Chemistry 1
Laboratory Manual

Fall 2008

Written by:
Dr. Maxine Davis
Dr. Ryan Sweeder
Dr. Steven Spees
Dr. Holly Bevsek

2

I. INTRODUCTION

Seeing is believing–this simple cliché is the reason why all good introductory chemistry courses have a laboratory component. There is nothing–no lecture, no photo, no simulation, no homework problem–that conveys the idea of "acid reacts with carbonates" as effectively as actually watching hydrochloric acid react with sodium carbonate. Here in Lyman Briggs we consider the laboratory to be an essential part of your education in chemistry, equal in status and well-connected to the lecture. Our hope is that you will find the laboratory interesting as well as useful for increasing your understanding of chemistry.

Helping you understand chemical principles is a very important purpose of the lab, however an equally important function of this course is to give you experience with a variety of techniques. These include: safe lab practices, keeping a laboratory notebook, proper handling of chemicals, performing dilutions, and using spectroscopy (the interaction of light with matter) analytically. Laboratory courses you will take in the future will assume you are proficient at these tasks so it is important for you to learn these now.

A final purpose of LBS 171L is to help develop your communication skills, which ranges all the way from how you work with a partner (partners should share work equitably; neither partner should have the burden of all the lab or writing work), to writing clear, concise descriptions of your work. This last point is very important when one considers that the ability to write well greatly improves one's chances of getting a scholarship, getting into a professional school, and getting a job.

Overall, through LBS 171L you will:

1. Demonstrate proficiency with basic laboratory techniques such as pipetting, preparing dilutions, and using spectrophotometry to analyze a solution's concentration.
2. Develop safe, proper laboratory working habits such as organization, cleanliness, and use of a laboratory notebook.
3. Record and present observations and other data in a scientifically acceptable fashion.
4. Present data analysis and discuss your results in a scientifically acceptable fashion.
5. Develop the ability to form hypotheses regarding chemical phenomena.
6. Design experiments to test these hypotheses.
7. Utilize chemical principles to analyze and evaluate these hypotheses.

Of course we, as instructors, have goals as well. Our goal is to provide a safe, collaborative, supportive environment in which you and the rest of your class can achieve the above goals and get the most out of your Briggs chemistry experience. Through some of the more open ended laboratories, we hope that you will also get a first-hand understanding of how science progresses.

Finally, we think chemistry and the chemistry laboratory in particular is fun! (Who doesn't like to see colored flames or solutions that change colors unexpectedly?) Although we can't promise that every minute of your laboratory experience will be greatly exciting–we don't know anyone who enjoys washing glassware–we do hope you will find LBS 171L enjoyable and useful.

II. HOW THE LAB WORKS

The laboratory periods are two hours and fifty minutes long. Most labs will begin with a short quiz covering the lab concepts from the previous week. Additionally a prelab exercise may be due if you are beginning a new laboratory that week. The prelab instructions will be in each week's lab. You will also be expected to write a brief purpose section in your lab notebook prior to lab (why are you doing this lab? What are you trying to figure out? What instruments/technique do you expect to be using to collect your data?) This portion of the prelab will be required for all labs, although it may not always be listed in the lab instructions.

The remaining time should be sufficient time to collect data and record all observations necessary to perform the lab assignment. Usually there will sometimes be time for you to perform any calculations or redo a part of the lab. If you finish early you are *strongly* encouraged to start your calculations in the laboratory while the experiment is fresh in your mind and your learning assistant is nearby. Each lab section will be supervised by two undergraduate learning assistant (LA) or one graduate teaching assistant (TA) who will be responsible for assisting the learning of and evaluating the students in their sections. Most of the LA's are Briggs students with outstanding academic and personal qualifications; their competence and enthusiasm for teaching makes them an invaluable resource for the Lyman Briggs learning community. Each LA will have one office hour which will be held in the chemistry help room. The LA office hours and room numbers will be posted on the course web page (http://www.msu.edu/course/lbs/171l/; http://Angel.msu.edu) no later than the second week of class. Feel free to seek help from any LA, the graduate assistants, or Dr Davis (davismax@msu.edu). Remember that your LA is a student like yourself and is also very busy with their own classes.

Safety

The chemistry lab contains many things which by themselves are not harmful, but through carelessness or inattention can become very dangerous. YOUR PRESENCE IN THE LABORATORY IMPLIES THAT YOU UNDERSTAND AND WILL FOLLOW THESE PRECAUTIONS:

1. Come to lab appropriately dressed. Clearly you will not want to wear your best clothes. Shorts, skirts, and sandals are absolutely forbidden in the laboratory (you may store a pair of sweats or running pants in your drawer to change into). Long hair must be tied back.
2. Contact lenses are not to be worn in the laboratory, even under your goggles. This is because solvents can diffuse through the contact and get trapped between the contact and your cornea. Save your eyes and wear glasses instead.

3. Absolutely no food, drinks, or gum are allowed in the laboratory. Smoking is prohibited (and just generally a bad idea for your health.)
4. All chemicals must be disposed of in their proper waste containers unless you are instructed otherwise by your LA. Spills or breakages should be reported to your LA immediately. Spills of solids or powders should be cleaned up using the dustpan and brush and then placed in a plastic bag and properly marked. Broken glassware should be deposited in the "broken glassware" container.
5. Report all accidents, no matter how minor, to your LA.

Safety Glasses:

Yes, this topic is important enough to warrant its own section. Eye protection is required at all times that you are in the laboratory when lab activities are being performed. Splash-proof goggles (the kind that completely cover your eyes and fit snugly on your face) are required for students. If you regularly wear glasses, you may request from your LA to be able to wear prescription impact resistant glasses. Any such glasses must have impact resistant side shields. The class will operate on a "**five strikes and you are out**" policy (what do you expect? This isn't baseball.) Your strike count accumulates over the entire semester, not just during one session.

Strike 1: A gentle private reminder to wear eye protection.
Strike 2: A harsh public order to put your goggles on, as well as a written copy of the Braille alphabet, because you'll obviously need an early start learning this.
Strike 3: Loss of all subjective evaluation points, and a visit to Lyman Briggs Safety coordinator Sue Rose. She will not be in a good mood.
Strike 4: Dismissal from the lab for the day, and a grade of "0" for the lab you were working on, with no opportunity for make-up work.
Strike 5: Failure of the course.

YOU ONLY GET TWO EYES. PRESERVE THEM.

Other safety rules

Your LA will give you specific safety instructions appropriate for each laboratory. Here are some general rules which you should be aware of for each lab:

$ Never handle/touch any solid or liquid chemical with your bare hands. Gloves are always available.
$ If any dangerous reagent splashes or spills on you or your clothing, wash it off immediately with plenty of water. For large-scale accidents remove all contaminated clothing and use the safety shower in the corner of the room by the periodic table. An eyewash station is also located by the shower–make sure you know where it is and how to use it.
$ Do not handle broken glassware with bare hands.
$ Always handle hot crucibles, beakers, etc. with a wire gauze or tongs.
$ Clean your work area when you are finished and wash your hands after finishing

the lab.

$ Do not use stressed, chipped or broken glassware. Give the damaged item to your LA and request a replacement.

Lab Notebook

You are required to have a lab notebook with fixed pages (not spiral bound), which is available at the University bookstore. In this notebook you should write a brief purpose for each lab along with all procedures, data, and observations. Always use pen (pencils smear easily) and make sure you write clearly so that it is legible when you analyze your data. If you make a mistake, record data incorrectly, or otherwise need to change something, draw one line through the entry you wish to change and record the correct entry the next line down, if appropriate. Do not erase or make the incorrect entry hard to read since you might need this seemingly "incorrect" reading. You need to have title on the first page of each lab (at the top of the page) and on every page you should have the date and page number. You should also maintain an up to date contents page at the front of the book. Twice during the semester you will be instructed to turn in your lab notebook so that your LA can evaluate it. During the first two weeks of lab, the LA will examine your notebook to ensure you are starting it correctly. Original data should be handwritten in the notebook, or if it is computer generated then the original data sheet should be taped in your lab notebook. It is also highly recommended that you write any conclusions or ideas that you come to regarding your work in the notebook. (This practice has saved the day in many patent law cases.) Remember that this book will be in the lab with you, so it is likely (probably expected) that it will pick up random stains and other signs of use. These kinds of stains will not affect your notebook grade.

III. LABORATORY REPORTS

As was stated earlier, being able to communicate well in writing is a critically important skill to learn. To help you develop this skill, you will usually have to turn in a lab report describing what you did and found in the laboratory. Some reports will be short, consisting of an abstract, your data, calculations, results, and any questions that might be posed in the laboratory manual. Most will be of medium length, consisting additionally of your procedure and a discussion of your results. Two will be full write-ups which will include an introduction. Although we recognize that this is more work than that done in the university general chemistry laboratory, we know you will reap the benefits from this experience in later classes (students who have taken this lab class before have stated that in later classes they felt they had an advantage over those who had little experience with writing scientific reports). See below for the expectations of each section. Keep in mind that the lab report is considered formal writing and you should write it as you would an English paper. That means full sentences, paragraphs with topic sentences and generally coherent thoughts. You should also avoid using the first person and contractions (two common mistakes) as well as having typos.

Policies Regarding Lab Reports

$ Lab reports are generally due the next lab section after you complete a laboratory–late reports will lose 1 grade level per portion of a day late. For

extenuating circumstances you must contact the instructor PRIOR to the lab.

$ Your lab report should be typed. It is strongly recommended that you *back-up* your lab report!!! Having to type up an entire lab report from memory in one night is not a pleasant scenario–backing up your files will prevent this from occurring (besides, no good scientist would ever leave their data on only one computer). Furthermore, there will be no special deadlines if your computer crashes or if your roommate deletes your files.

A typical lab report will include many of the following pieces:

$ Title Page
$ Abstract
$ Introduction (if required)
$ Procedure (if required)
$ Data
$ Calculations
$ Results
$ Discussion (if required)
$ Questions (if required)
$ Conclusion (if required)

Components of the Lab Report

Page Numbers: Insert page numbers on each report

Title Page: The title page should have a title for the experiment, the date you performed the experiment, your name, your partner's name if applicable, your LA's name, and section number.

Abstract: An abstract is a brief summary of your study. It states what was studied, what method was used to study it, and what the results (including standard deviations) were. With the enormous volume of scientific papers published, abstracts are necessary to convey the essence of your study to a person who does not have time to thoroughly read your report. Keep in mind that reports are not mysteries. You are allowed to give away the ending in the first paragraph. Rarely will an abstract exceed 200 words (they are usually much shorter.)

Introduction: The introduction is the section where you should provide the necessary background for the reader to understand your lab. This may include a bit of contextual information (what was the general purpose/goal or why should we care) but it should also contain the necessary *chemical* background information. This section is what allows us to really get feel of if you understand the chemistry or not. So if the lab involves heavily upon redox chemistry, then you should remind the reader of what oxidation and reduction is. This section will usually run at least a page and less than about four. Of course the length depends heavily on what you are writing about. Remember that you are trying to

provide enough background so that the discussion will make sense.

Procedure: A procedure is the description of your scientific work in such a manner that the experiment can be independently repeated and verified. As such, the amount of detail of your procedure should be such that someone would be able to carry out the laboratory on their own. Remember though that this is still a report of what you did, NOT instructions on how someone else should do it. Thus you must keep good notes in your lab book so you can write up exactly what you did. Since you have done this in the past, your lab report should be written in the past tense (remember to avoid the 1st person too.) In writing your procedure many steps will be implied. For example saying you dissolve 4.20 g of NaCl in 40.02 mL of water implies that you carefully measured both the quantity of salt and water. Rarely will you need to be specific in the type of glassware that was used (unless you used something highly unusual). Cleanliness is ALWAYS expected and so need not be explicitly mentioned.

Data: All experimental measurements, observations, and results should be *tabulated* in a "data" section. Units should be included for every quantity and follow guidelines for significant figures (your textbook talks about significant figures if you do not remember them.) Follow the guidelines for creating tables found below. Strive to keep the number of tables to a minimum by combining different tables where possible (this usually makes it easier to read too.) [I will note that this section is often not explicitly included in many scientific articles, but the necessary data is usually provided somewhere. This simply makes the LA job a little easier.]

Calculations: This section is necessary for your LA and instructor to follow your work. Show one example of every kind of calculation (even unit conversions and standard deviation) and remember to use the correct number of significant figures. [Again, this is not usually in a scientific article, but this process makes it much easier for us to identify possible errors in your methodology for determining results.]

Results/Discussion: In these sections (which are often combined into one), you should summarize what results you obtained in your experiment and provide the logic for how you reached your conclusions. When you discuss your results, remember to mention applicable theories or hypotheses you might have initially made. Remember that your discussion should be based on the results you *actually* obtained, not the results that you think you should have obtained. Overall, this section helps you take the data that you collect, and using the information provided in the introduction, shows the logic of how you reach the conclusions. It is also wise to discuss any sources of error, general problems with the method, improvement, and/or future directions for research as appropriate. If you discovered that your entire method failed because of some error, that is okay. Point it out clearly and explain WHY your method failed and how you could have improved it. (Oddly such lab reports are often some of the best.) Remember you are being evaluated based on your reasoning skills and logical processes more than the specific results.

Questions: There may be a few questions at the end of the laboratory. Whenever possible, it is recommended that you address these in the discussion section (as usually they are good discussion fodder.) If it makes no sense to do it there, you may need to have a specific "Question" section. Be sure to include any necessary calculations (if they are not in your calculation section.

Conclusion: This is the final section and is exactly what it sounds like. You should be summarizing what you did and your findings that you just presented in your discussion. Also including any next direction to go based on your findings is appropriate. Often this section looks very much like your abstract.

Text and all data submitted are expected to show professionalism in both presentation and grammar; points may be deducted otherwise. This should be considered a formal English paper afterall. You are also expected to cite any reference materials used and give credit to any other students with whom you worked. There is no specific formatting necessary for this, though chemists will typically use numerated end notes for their citation. Look at the website for more information on citations **Copying part or all of another student's report will minimally earn you a zero for that laboratory; more severe repercussions (failure in the lab, expulsion) will result for repeated or egregious cases of plagiarism.**

IV. GRADING

Lab reports will be graded in a holistic manner. That is that they will receive a single overall grade. Formal lab reports will be given twice the weight of the smaller reports in the final course grade. Each lab report will be given one of the following grades (expected quality suggested afterward):

4.0 – Outstanding report – a basically perfect report with no significant errors
3.7 – Excellent – a few minor mistakes no significant errors
3.3 – Very Good – one or two small errors
3.0 – Good – a well written/understandable report at effectively communicates ideas. Some errors present.
2.5 – Fair – writing or conceptual errors present, but the general idea is correct
2.0 – Minimally acceptable – significant errors present. Some effort shown.
1.0 – Poor – At least you handed in something with some concepts present.
0.0 – Did you not show up?

The LAs will grade all lab reports. A randomly selected subset of lab reports will also be graded by Professors Davis and the graduate TAs.

There will also be prelab assignments. These assignments will be simple activities worth 5 points each. They are designed to get you to read the lab before you show up. Reading the lab will not only help you do the prelab assignment, but also will allow you to get through the lab quicker. There will also be post-lab quizzes the week after each lab. These quizzes will be designed to determine if you really understood what you did in the lab. If you understood the lab well enough to write a good lab report, you will have no problem with these. If you basically copied a lab report from someone else,

9

expect to have difficulty with these. The moral---ask questions if you don't think you are understanding the lab. These quizzes will be worth 10 points each.

There are also 60 subjective points for the semester. Your LA will evaluate your performance in lab each week and assign these points as appropriate (for example, a loss of points could result from not reading the laboratory beforehand, not cleaning your lab bench, from using poor technique, or using poor laboratory safety practices). Points will be deducted from the entire class if common areas are left messy (this is by far the most common reason to lose points.) Notebook collection will be worth 20 points.

Grades are ultimately decided by the laboratory coordinator, Drs. Davis. Grading differences on lab reports among LAs will be accounted for by standardizing everyone's grades at the end of the semester by comparison of the reports graded by the instructor. Thus if your LA is grading harder than the professor, not only will you hopefully be getting better feedback, but you will also have an increase if you lab report grade at the end of the semester. If they are grading easier,… then it may be best to come talk to the professors. Last year lab reports accounted for approximately two-thirds of the total grade with the other third coming from all point-based grades (prelab, quizzes, participation, notebook collection.)

V. FIGURES, GRAPHS AND TABLES

You will make quite a few figures, graphs and tables. Here are some guidelines:

- Each figure, graph or table should have a title. Graph titles are always numbered and state the dependent variable versus the independent variable, or "y vs. x", i.e., *Graph 1. Absorption vs. Concentration* (see example below). Tables should also always be numbered and have titles (see below). Figures should be numbered and have titles, but the name and title should appear below the figure itself.
- Graphs and tables should always have correct units and significant figures on them.
- Do not play "connect the dots" with your data points on a graph. Usually you will have to perform a least squares analysis (Excel does this easily) to obtain a trendline which shows the general pattern of your data.
- Label the ordinate and abscissa axes (the y and x axes), including units.
- On graphs, choose your axis scale such that any trend is clear. You do not want a scale so large the data are scrunched together; you also do not want a scale so small your data appear randomly scattered.

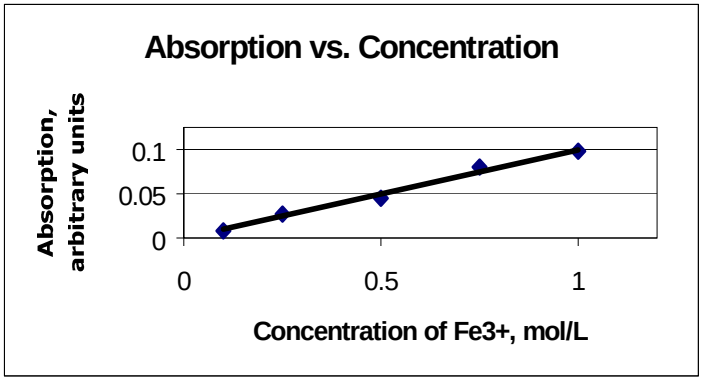
Here is a sample data table:

10

Table 1. Absorption of various [Fe^{3+}] solutions at 540 nm.

Concentration of Fe^{3+}, mol/L	Absorption, arbitrary units
0.100	0.008
0.250	0.027
0.500	0.045
0.750	0.080
1.00	0.098
Unknown	0.031

Here is a sample graph from the above data:



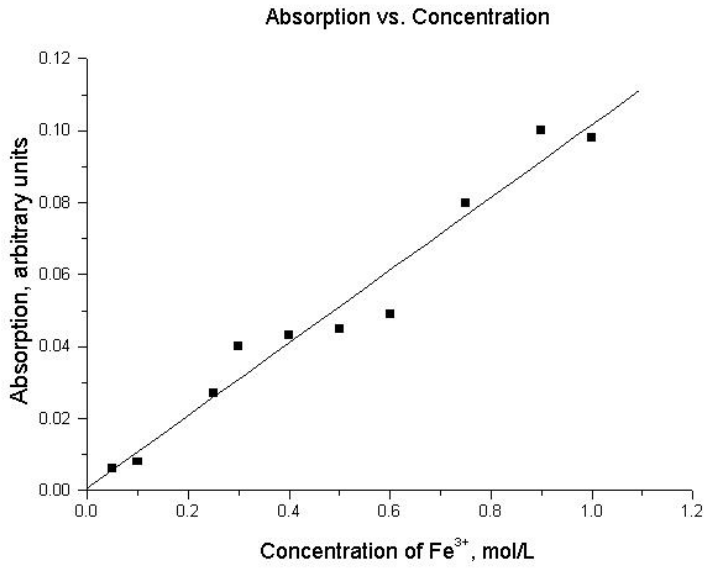


Figure 1a. Accurate but imprecise data.

11

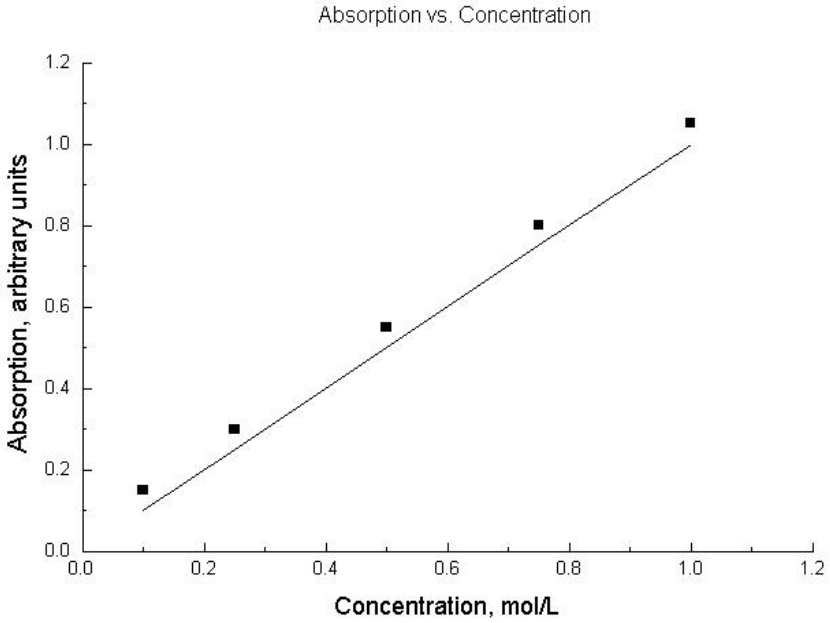


Figure 1b. Precise but inaccurate data.

VI. ACCURACY AND PRECISION

The *accuracy* of an experiment is a measure of how close the result of the experiment comes to the "true" value. Of course, in most experiments we do not know what the "true" value is; this makes accuracy determinations difficult at times. *Precision* is a measure of how well the result has been measured, independent of how closely the measurement agrees with a "true" value. It also is a measure of how reproducible the experiment is. Figures 1a and 1b below show the difference between accuracy and precision.

Exact numbers

Exact numbers are those that have defined values or are integers that result from counting numbers. Examples are 1 m = 1000 mm, 2.54 cm = 1 in., and 4 wheels on a car.

Inexact numbers

Numbers obtained by measurement are always inexact because error is inherent in every measurement. Human error results from mistakes in measurement or computation; they are usually apparent either as obviously incorrect data or as results that are not reasonably close to expected values. Situations causing human error include trying to measure 50 mL of a liquid in a graduated cylinder and not drying the cylinder out beforehand, or using an incorrect conversion factor. These kinds of errors SHOULD be easy to eliminate. Having bad data to inexact numbers typically indicates carelessness/thoughtlessness in lab.

Systematic errors affect the accuracy of an experiment—they make results different from the "true" values with reproducible discrepancies. They can result from faulty calibration of an instrument (note that this is not a human error) or personal bias. Discovering sources of systematic error is an important task of the scientist and is not always obvious. When planning an experiment, spend time considering ways to reduce the sources of systematic error. Avoiding these are critical, though it is often challenging to eliminate them. That of course is the ideal goal.

Random errors affect the precision of an experiment. These errors are the reason why experiments may yield slightly different results from day to day or from lab to lab. Reducing these errors usually means repeating the experiment to obtain better statistics, but using more precise instruments or a more refined technique are other ways to reduce random errors. You should always repeat any experiment if possible. It is usually impossible to make any meaningful conclusion based off of a single data point. Random errors are typical and should be expected. They are simply a part of science.

VII. SIGNIFICANT FIGURES

Measurements are generally reported in such a way that only the last digit is uncertain. All digits including the uncertain one are called significant figures (another way to think of this is that the number of significant figures is "every one you can read plus one you guess at"). The numbers of significant figures indicates the exactness of the measurement. A measurement of 3748.952 has seven significant figures and the uncertainty of the measurement is ±0.001. This is a critical concept in lab. There is a very notable difference between the meaning of 4 mL of a liquid and 4.000 mL of a liquid.

Math and significant numbers

The precision of the result is limited by the precision of the measurements. Thus, it is important to use the proper amount of significant figures in the results of calculations. In multiplication and division the result must be reported with the same number of significant figures as the measurement with the fewest significant figures (remember that converting a fraction to a number is division). In addition and subtraction the result cannot have more digits to the right of the decimal point than any of the original numbers. Your textbook has a discussion of significant figures in sections 1.7; refer to these if you would like a review of significant figures. In lab, ask the LAs about the level of precision available from any kind of glassware.