CHEMISTRY 120

Laboratory Manual Preface

last updated 2/2005

Thomas P. Beebe, Jr.

Quantitative Chemical Analysis II

Chemistry 120
University of Delaware
Department of Chemistry & Biochemistry

Laboratory Safety Guidelines

1.) Know the location and use of fire extinguishers, fire blankets, fire exits, safety showers, eye wash fountains, first aid kits and chemical spill kits.

2.) Wear safety goggles at all times. The safety goggles must be worn over your eyes and not on your forehead!

3.) Wear sensible clothing. No open-toe shoes or sandals. Keep long hair tied back. Use protective clothes and gloves when handling dangerous chemicals.

4.) Do not smoke, eat or drink in the labs.

5.) Prevent cuts and burns: Discard chipped or broken glassware immediately. Be aware of hot objects. Never heat volumetric glassware.

6.) Immediately report any and all accidents to the lab assistant. This includes small cuts and burns.

7.) Be cautious of others. They can be your biggest safety hazard.

8.) Avoid all flames if flammable solvents are used nearby. Those using flammable solvents must inform the lab assistant and work in the hood.

9.) Never pipet by mouth. Use a pipet bulb.

10.) Never conduct unauthorized experiments. Severe penalties will be imposed for this.

11.) Never work alone in the lab. Lab assistants may give permission for valid excuses for students to make up missed lab periods only when pre-arranged with the lab assistant. In doing so, the lab assistant agrees to be present at all times. This can only go on during the regularly scheduled lab times.

12.) Follow all recommended safety precautions. If you miss the prelab, consult the lab assistant before beginning your experiment.

13.) Keep all toxic and volatile materials in the fume hood.

14.) Keep a neat and clean lab. Label all containers.

15.) Dispose of chemicals only in the proper container, as instructed by your lab assistant. Chemical disposal issues can not be taken lightly. The university faces severe fines for improper chemical disposal (i.e. putting materials down the drain, putting chemicals into waste containers without the proper labels, or putting the wrong chemicals together).

16.) **THINK !!!**

Please sign the following form, indicating that you have read these safety rules and agree to abide by them during the period of this course. Tear out and turn in your signed copy to your lab assistant before the first class.

Safety Agreement

I have read the safety rules for this course and I understand them. I agree to abide by them to the best of my ability and to do my best to make the lab a safe place to work.

Name: _____

Date: _____

Phone: _____

E-mail*: _____

****If you use a non-UD e-mail account such as Hotmail or Yahoo, make sure that you have your official UD e-mail forwarded to that account. The UD shotgun e-mail lists can only use your official UD e-mail account. We will make extensive use of e-mail to communicate in this course since there is no lecture when we all come together.***

Laboratory Procedures and Policies

1. All laboratory operations must be performed during the lab section for which you are registered.
2. If a legitimate emergency arises (a death in the family, illness, etc.), contact your regular TA in person or by phone or by e-mail BEFORE THE ABSENCE and inform him/her of your intended absence.
3. It is the responsibility of the student to schedule his or her own make-up lab, in consultation with the TA.
4. All students are responsible for making sure that their instrument and the surrounding areas are left in good working order, and that it is as clean or cleaner than the condition in which you found it.
5. Report a dirty or broken instrument to the TA as soon as you are aware of this problem.

Safety Considerations and Right to Know

In addition to the safety issues raised above, you should assume that all chemicals used in the labs are dangerous and harmful. Treat them as such, and be aware of their toxicity. You have a RIGHT TO KNOW what chemicals you are using, and what their toxicity is. To this end, each chemical that you will use in the labs will have a Material Safety Data Sheet (MSDS), located in a 3-ring binder on the shelf. These sheets are useful to provide information about safe handling procedures, acceptable exposure limits, treatment procedures for excessive exposures (especially for emergency responders and medical staff).

Laboratory Notebook (10 %)

A laboratory notebook should provide a permanent record of details in procedure, raw data, observations, calculations and results. The criterion for sound record keeping is that someone else should be able to readily locate and understand the pertinent results for an experiment. Although reasonable legibility and neatness are required to meet this criterion, the utility of a record is determined largely by whether it is original, systematic and complete, not by whether it is a work of art. So don't get hung up on neatness to the level of compulsion. The following are guidelines for keeping a proper experimental record:

1. A <u>bound</u> notebook is required for this lab. Numbered pages are preferable. Please do not choose a spiral-style binding. Several other styles are available in the bookstore. Please buy one and bring it to the first and each subsequent lab period. You may also use a lab notebook from a previous course if there is enough room left.
2. Enter all data in ink as soon as it is taken. Never recopy numbers or use loose sheets of paper. Cancel errors or rejected data by drawing a <u>single</u> line through them, ~~like this~~. Do not erase or remove pages. Set aside the first two pages of the notebook for a table of contents which you will organize at the end of the course.
3. Enter data only on the right page of the two facing pages. Use the left side as a scratch sheet for calculations.

4. Clearly label all entries, including the units (*e.g.* g for grams, mL for milliliters). Don't forget to record your unknown number, and date each page.
5. Even if calculation of final results from the data taken in lab is done at a later time, record these calculations in your notebook for future reference. To avoid errors in calculation, write out the balanced chemical equations for the reactions carried out in the determinations.
6. Make a record of all computer-generated printouts in your notebook, and carefully notate each page of printout so that it is clear what sample/standard/experiment it goes with.
7. Bring your own floppy disk to lab each time so that you can make a copy of data to take with you.
8. Before leaving the lab, have your TA initial your lab notebook. You will be graded on this aspect of your work (10% of the lab grade).

The Pre-Lab (30 %)

Before you come to the lab, study the lab you are about to perform, and set up your notebook. At the beginning of the section for the lab in question, write a **Summary** of the goals and methods of the experiments. Do not simply copy from the lab – use your own words. This is usually a two- to three-sentence paraghraph. Write out a step-by-step **Procedure** for what you will do in the lab. To facilitate direct recording of experimental work, also set up **Data Pages** and **Data Tables** for each experiment **before coming to the lab**. Labelling a space for each piece of data you must collect will ensure that no step is overlooked. Your TA will discuss with you some of the aspects of the lab you are about to perform. He or she will quiz you about certain things that you should know before you start your lab work. You will be graded on all of these aspects of preparation called the **Pre-lab** (30% of the lab grade).

The Laboratory Report (60 %)

Although a detailed and specific format is given below for the Lab Report, it does not imply that your Lab Report should be a 10-hour masterpiece. You must be the judge of how much time to budget for the Lab Report. I would recommend that you not spend more than 2 to 3 hours on the write up each week. Try to find the right balance between a report that is well written, well organized, and yet not so detailed that it takes you an inordinate amount of time. Do not exceed the page limits stated below.

A specific format is required for all Chem 120 Lab Reports. **Be sure to follow it carefully so as not to lose points.** Use the numbered section headings when preparing your reports. The reports shall be printed using word processing and spreadsheet software. All graphs and charts shall be prepared using this software and embedded in the body of your report. Use one-inch margins on all sides and Times New Roman 12-point font, double-spaced. All pages should be numbered.

Format of the Laboratory Report

Title Page. Give the title of your project, your name, your partners' names (if any), the date, *etc*.

Abstract. This is a brief one-paragraph summary of the main findings, the technique used, and the conclusions. Look at abstracts from a current issue of the journal *Analytical Chemistry* to get an idea of the style used in an abstract.

I. Introduction and Purpose. State the objectives of the project. Specify what information about the sample is being sought and the significance of obtaining that information. Describe in *general terms* the methods you have used to obtain the specified information: summarize the theory of the chemistry employed including any formulas or equations required to calculate the result. ***This section should be no longer than 0.5 to 1.0 page at most.***

II. Experimental. A *very brief outline* or statement of the procedure should be included. List all steps involved: sampling, sample preparation, isolation of the analyte, the measurement step, and standardization. Describe any difficulties that you encountered or any tricky steps in the procedure. A brief sketch of any special apparatus required is useful for later reference. **Do not describe the analysis by copying verbatim from the lab write-up, and do not use a "cookbook recipe" style!** An example of a cookbook style is: 1.) Add 1.2 grams of compound; 2.) Heat until dissolved; ... Instead, use proper scientific writing style which you can find in any research journal. Examples of a proper scientific style for the above steps are: 1.) 1.2 grams of compound were added…; 2.) The sample was heated until it dissolved completely; … ***This section should be no longer than 0.5 to 1.0 page at most.***

III. Data. Organize the raw data that you have collected into tables and graphs where appropriate. Computer spreadsheet programs such as Excel are very helpful for this, and their use is encouraged for tables and graphs. Incorporate such tables and graphs directly in the body of the text. Do not copy or paste the original computer-generated output here; put such items in the **Appendix**. ***This section should be no longer than 1.0 page at most.***

IV. Calculations and Results. Pay close attention to avoiding calculation errors, since these can ruin the most carefully gathered data. One example of each type of calculation should be given, along with the equations used. MS Word has an equation editor that you should learn to use if you have not already done so (/INSERT/OBJECT/ SCROLL DOWN TO MICROSOFT EQUATION 3.0). Tabulate the final results for any standards and the unknown samples. ***Keep this section as short as possible (0.5 to 1.0 page at most)***.

V. Error Analysis. Calculate the means and standard deviations of replicate results and perform *t*-tests and *F*-tests if appropriate (see chapters 3 and 4 of Harris text). All reports should include a qualitative description of the sources of error in your measurements. In addition, when possible, use **propagation of error analysis** to make a more quantitative assessment of the errors in your reported results. Linear regression plots should report the slope and its standard error, and the intercept and its standard error. These are generated in, for example, the Excel linear regression output. ***Keep this section as short as possible (one page or less)***.

VI. Discussion. From the statistical analysis data, discuss the significance of the results that you obtained. Compare them with any known or expected values. Consider whether the precision of your results is reasonable for the particular method of analysis. Describe any potential sources of determinate or systematic error which your standardization did not correct. Propose any steps which might be taken to improve the method. Normally this section would not include comments about the method since the method is assumed to be fine for what you want to determine. ***This is the section where questions raised in the lab manual should be addressed. This section should be one page or less.***

VII. Conclusions. State the conclusions of your project. Be concise.

VIII. References. Self explanatory; use the format found in the American Chemical Society journal *Analytical Chemistry*, and include all titles.

IX. Appendix. Include copies of notebook pages, computer-generated instrument output and other data not appropriate for the body of the report.

How and When to Submit Your Lab Report

Your report should be turned in ***no later than one week following the lab period that you finished the lab***. The report should be turned in directly to your TA, at the ***beginning*** of the lab period. When the due date falls on a day that the university of closed, your report is due on the next day on which the university is open for business, by 4:00 PM, in your TA's mailbox. Do not turn in reports to anyone other than your TA. A penalty of 5% of the total available points will be assessed per calendar day for late reports.

Working in Teams

You will generally work alone, or with one other stufent on each lab experiment. Each team member is expected to contribute equally to the lab work and to the written work. When you have worked with someone else on a lab, this does not mean that you can copy their write-up and hand it in as your own. All team members are expected to be present for all parts of the experiments. You may not divide up the tasks and go away while your partners do their parts. All team members must turn in their own unique Lab Report, although the calculation of the results can and should be done working together. Do not prepare a single report for the team and make several photocopies of it. **Each student must write his or her own report.** Set up a time when you will get together with your lab partners to do the lab calculations and discuss the results.

Recalculation of Results

When a failing grade is obtained on a lab, it is important to attempt to locate the error as soon as possible so that similar errors can be avoided in the future. The lab assistant can help in this manner, but it is better if the student can identify the error. Some failing results are caused by calculation errors using perfectly fine data. Common examples are the use of incorrect formulas, misplaced decimal points, omission of dilution factors, too few or too many significant figures, and missing or incorrect units. Most of these can be avoided by diligent record keeping in the lab notebook. Poor agreement between replicate measurements is often a signal that something is wrong with your technique. If you have difficulty locating the problem, take your notebook to the lab instructor and discuss the situation.

A student reporting poor results which are caused by incorrect calculations will be given a returned report marked "Recalculate". The student may recalculate results from the original data to receive full credit, minus a 15% penalty for the calculation error. In this case, the student must return his or her recalculated report within 7 calendar days, and he/she must clearly demonstrate that only the calculations are new, not the data! Your notebook will be examined and compared with the copies of your notebook pages handed in originally with the lab. **This is not an opportunity to make up better data !!!**