

Chemistry 341

Physical Chemistry Laboratory

The purpose of the laboratory component of the course is several-fold. It reinforces the material you have learned in class and it gives you a chance to apply your knowledge. You will learn some important experimental techniques that are necessary for you to become an effective chemist. The lab will allow you to delve more deeply in some topics and perhaps to cover others that we will not have time to touch upon during class. It will require you to learn how to deal with complexity, both in experimentation and data analysis, which is an important component of any scientist's expertise. Two of the lab reports first semester are to be given orally. Oral reports are aids to you in learning how to express your scientific ideas verbally, something you will need to do the rest of your life.

The most important component of completing a laboratory successfully is your pre-lab preparation. You will make fewer mistakes if you have read and understood the lab write-up. In many cases you will need to figure out how to make up the necessary solutions for the lab before coming to the lab; there isn't enough time for these calculations during the lab. A half-hour of time spent before the lab may save hours on Monday (or Tuesday) afternoon. Short quizzes at the start of some of the labs will help you to remember to prepare. You may use your lab notebook for these quizzes, but not the lab write-up. You must change lab partners each week.

The first week of lab covers error analysis and least-mean-squares curve fitting, which requires the use of the computer. This assignment will help to refresh your spreadsheet skills. These techniques will be used repeatedly throughout the year.

Laboratory reports are due one and a half weeks after completion of the experiment (on Wednesday for the Monday lab or Thursday for the Tuesday lab). Late lab reports will not be accepted more than two weeks late. All lab reports are due before the final. Oral reports will be scheduled during the week following the corresponding labs. Two of your lab reports will be written, two oral and for three you will just hand in the calculations and answers to questions. For the two written or two oral labs, you may, if you like, turn in your calculations early to be checked for problems. In this way, calculation errors can be corrected before you turn in your written or oral labs! You turn in your trial calculations on the special report sheets kept in the "Quick Check Calculation Sheets" section of the lab Web page, no later than the Friday of the week you complete the lab. We will be more than happy to help you with any problems that come up in writing up your labs, calculations included.

You will be required to complete 7 laboratories, which includes the initial pK_a of methyl red lab. Some of the labs are paired, that is, the two labs complement each other. These labs are 10 and 11 on keto-enol tautomerization and labs 12 and 16 on the acetone-chloroform system. Labs 17 and 18 on Solid-Liquid phase equilibria also make a good pair, for Geologists. Chemistry is an experimental science, the key to which is the ability to verify our theories of the processes in the physical world. Have fun probing nature.

Lab Reports

The goal of this course is to help build your independence. The calculations in physical chemistry can become complex. So, at first you may need to develop the mathematical skills to be successful with complex calculations. To ease your way, the first few lab reports will be done collaboratively with your lab partner. Please follow the following schedule. When collaborative

work is done, turn in just one copy under both partner's names. When you aren't collaborating on calculations, you will work independently after you finish your measurements.

For the written labs, only the first is a full lab report with Introduction, Theory, Procedure, Results, and Discussion. For the second written lab in week 6, for the Theory and Procedure sections just reference the lab write-up and briefly mention any changes in procedure from the lab write-up. In other words, for the short-form report, the Theory and Procedure sections are just references (e.g.: please see CH341 Lab Manual Experiment 3 for the theory and procedure) and usually nothing else. For your oral reports have all the tables, graphs, and calculations in your lab notebook. Your discussion and error analysis will be given orally, but you can make notes in your lab book. For the oral labs, please also fill out a report sheet, kept in the "Quick Check Calculation Sheets" section on lab Web page.

Full and Short Form Introduction: In the Introduction, describe the purpose of the experiment and the expected result in a few sentences. For example, the purpose might be to "do a kinetics study for a specific compound" and the expected results might be "the rate constant for the reaction at several temperatures and the activation energy." Lab reports need not be typed.

Full and Short Form Theory: For the Theory section, just reference the write-up, but include any requested derivations.

Full Procedure: For the Procedure section for the first lab report, describe enough of your procedures so that another student could easily repeat your experiments. Tell exactly what you did using explicit volumes, weights, and temperatures. Use past tense to describe your procedure. Don't copy the procedure from the write-up; state exactly what *you* did.

Results for all Reports, Oral and Written: For the Results section, provide the data in a tabular format, including **all information necessary to repeat your calculations**. Columns in your tables should have easily understood headings and include units. Attach all your graphs. Graphs should fill at least a third of a page and include clearly labeled axes including units. (Axes labels and units, etc. can be hand written.) Include uncertainties for all derived values (see the error analysis handout for instructions for representing uncertainties). For example, slopes and intercepts from curve fitting should always be given with uncertainties. Remember to use significant figure rules in presenting the final results.

Discussion for Reports, Oral and Written, Full and Short Form: In the Discussion section, discuss the chemical significance of the results. In other words, state why these results are useful and important. State how this experiment and technique fit into the larger world of chemistry. Discuss why someone might need to do a study of this type. Are the results for this system unusual or do they fall within the normal range for other systems? Answer any questions in the calculations and discussion section of the lab write-up. Also comment on the uncertainty of the final results: what are the predominate experimental errors? Compare your final results to literature values, if available from the first listed reference, Atkins appendices, Lange's Handbook, or the CRC. Make sure to give a citation for the literature values or any other information that you obtain from literature sources.

Calculations Only Reports: For three of the labs you will hand in your calculations and the answers to any questions from the lab manual. A discussion section is not otherwise required.

Provide the data in a tabular format, including **all information necessary to repeat your calculations**. Attach all your graphs. Include uncertainties for all derived values (see the error analysis handout for instructions for representing uncertainties). For example, slopes and intercepts from curve fitting should always be given with uncertainties. Remember to use significant figure rules in presenting the final results (keep a couple of significant figures for intermediate results to avoid round-off error and help in checking the accuracy of your calculations).

Computational Labs: There are no lab reports for the computational labs. For the computational labs you just hand in the answers to the questions in the exercise write-up. You answer these questions independently. In other words, the computational labs are just like homework. The computational labs are the least means squares curve fitting assignment and the exercises done in the Schupf lab from the “Molecular Mechanics Tutorial” and the “Computer Aided Molecular Design Tutorial.”

Lab Report Schedule (non-computational labs)

Lab	Report format	Collaboration
1 pKa Methyl-Red	written- full form	collaborate
2	calculations and answers to questions*	collaborate
3	calculations and answers to questions*	collaborate
4	oral	collaborate on calculations
5	oral	independent
6 Equilibrium	written- short-form	independent
7 Solubility of AgBrO ₃	calculations and answers to questions	independent

* This lab is graded all or nothing: your calculations must be correct for credit and only full credit will be given. You may submit your calculations as many times as necessary. Calculations must be turned in directly with both partners present. The instructor will check your calculations while you wait. This procedure will guarantee fast turn-around times for grading and provide timely answers to questions when information is missing from the report.

RULES OF THE ROAD

PREPARATION

1. Read carefully and understand the lab write-up before coming to lab.
2. If you don't understand something--ask. Labs are a lot of fun, if you are prepared.
3. Look up all of the substances in the experiment in the **Merck Index** (or other suitable source) to determine if there are any health or safety hazards.
4. If you are doing a synthesis, look up the reagents in Fieser & Fieser, "Reagents for Organic Synthesis", to determine if there are any hazards, purification instructions or other helpful tips.
5. Plan out your solution preparation scheme before coming to lab.
6. Write everything down in your lab book.

IN THE LAB -GENERAL

1. Wear eye protection (goggles or glasses with side shields).
2. Note the location of safety equipment, fire alarms, and exits.
3. Be conscious of what others are doing around you.
4. Clean up chemical spills immediately, especially in and around balances.
5. Check with the instructor for disposal information on all chemicals and solutions. Unless stated otherwise collect all waste in labeled waste containers. Keep aqueous and nonaqueous waste separate.
6. Never bring any library books, journals, reference books, etc. into the lab. Many people need to use these materials, they must be kept in good condition. Keep track of important information in your lab book instead.
7. Never heat volumetric glassware on a hot plate. Don't store solutions in volumetric flasks. If you get a solution into a pipet aid--wash it out immediately.
8. Check with the instructor for the proper procedure for washing spectrophotometer cuvettes and cells. Never wipe cell windows with paper towels.
9. Work with concentrated acids or bases in the hoods only.
10. Make up solutions in the P/Q lab, not in the Instrument lab.
11. Weigh out chemicals by difference or into small beakers. Don't use paper for weighing.
12. Never place a pipet directly into a solvent or solution bottle. Pour just what you need into a small beaker and pipet from the beaker.
13. Never return reagents to the bottle.
14. Record everything in your lab notebooks.
15. You will often work in pairs. Both members of the pair must be present throughout the course of the experiment.

CHEMICAL GRADES

Chemicals, especially solvents, come in many different purities or grades. Make sure to use the grade best suited to your experiment.

1. Technical grade solvents or chemicals are usually not pure enough for P. Chem or Quant. experiments. Generally use reagent grade for this purpose.
2. Don't use "Primary Standard" or ultra high ("Ultrex") purity reagents unless specifically indicated in the write-up.
3. Don't use "Spectro" quality or "HPLC" grade solvents unless specifically indicated.
4. Never place a pipet directly into a solvent bottle.
5. Never return reagents to the bottle.
6. Always filter HPLC solvents, buffers and samples using 0.2 micron filters.
7. Laboratory water comes in different grades, too. Think about the grade of water that you need to use. Distilled, deionized and high purity (grade 1 "reagent grade") water are available. Laboratory water is expensive--use it sparingly. In rinsing glassware, several rinses with small volumes of water are much more efficient than one rinse with a large amount of water.

All of the pointers above may seem bewildering at first, but they rapidly become automatic. Mastering good laboratory techniques will make you more confident, which means that lab will be a lot more fun and you will be able to exercise your creativity to a greater degree.