

CHEMISTRY 4620
PHYSICAL CHEMISTRY LABORATORY

Syllabus Spring 2017

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NO required text; *lab packet of experiments will be distributed.* *Lab notebook is required*

Week 1: Keeping a Laboratory Notebook.

The recording of not only data, but nonstandard procedures and observations of unexpected phenomena are critical to understanding of scientific research. In a way, the laboratory notebook is a journal of what happened during the course of running an experiment. One must always leave open for the possibility of serendipity (often the result of 'mistakes'), which leads to new and important discoveries. Detail is extremely important. During this first lab period, we will go over an NIH sponsored webinar by Dr. Philip Ryan of the Office of Intramural Training and Education, entitled 'Keeping a Lab Notebook.' This webinar can also be found at [https://www.training.nih.gov/assets/Lab_Notebook_508_\(new\).pdf](https://www.training.nih.gov/assets/Lab_Notebook_508_(new).pdf) for future reference. The lab notebook will be weighted as one laboratory report. It will be assumed that students are recording all pertinent data and relevant information and observations. At the end of the semester, notebooks will be turned in for evaluation.

Course Structure

The physical chemistry laboratory will meet once a week, on Tuesdays from 9:00 AM -12:50 PM in 439 NE. Each lab session will usually consist of a brief introduction/comments, and then students will go into the lab to work. **IT IS ASSUMED THAT STUDENTS HAVE READ AND UNDERSTAND THE LABORATORY BEFORE COMING TO CLASS.** Anything you do not understand you should bring up during the introductory lecture. *Students will work in assigned groups.* Since the experiment performed during each lab section will vary from group to group, a schedule of experiments according to group will be given.

List of 7 Experiments

Dynamics: Viscosity, Conductivity and Kinetics

1. Gas Viscosity* Handout includes the experiment set-up and the calculations to be carried out on your data. This is followed by a section on the Kinetic Theory of Transport, which you should read. Skip the section on Effusion of Gases. Read Gas Viscosity. Another version of this experiment, Experiment 4, is included for reference.
2. Conductance of Solutions (Note: You will use a conductivity meter, so do not read about the discussion of the Wheatstone bridge).
3. Kinetics of EDTA binding

Introduction to Spectroscopy

4. Absorption Spectrum of a Conjugated Dye
5. Absorption Spectrum of I₂ (skip Emission)
6. Vibrational Spectroscopy of CO₂
7. Gouy Balance*

*The report for these experiments is just a worksheet, not a formal report. However, they count as a report for the purposes of the schedule on the previous page.

Weeks 10-13: Special Project-to be determined

Week 14: Oral presentation

Special Project: During the last class meeting, each student will make an oral report of their special project, approximately 15 mins each. A print out of the presentation must also be turned in.

Lab Report Due Dates

Report Number	Due Date
#1	Feb. 21
#2	March 14
#3	March 14
#4	April 4
#5	April 4
#6	May 2
#7	May 2

Reports must be submitted in hardcopy – e-mailed lab reports will not be accepted.

Format of Laboratory Reports

RULE 1: Be concise; longer is not better.

Laboratory reports will consist of the following parts:

- I. **Title of the experiment**, the name of the student and his/her partners, and the dates the experiments were completed.
- II. **Objective.** 1-2 sentences clearly and specifically stating the point of the experiment. What will you be calculating or determining?
- III. **Method.** Not needed.
- IV. **Raw Data.** Raw data is organized in tables or graphs. Present data clearly. All units are included and titles are included. Title example: Plot of fluorescence intensity (counts) as a function of wavelength (nm) for 3-methylindole with an excitation wavelength of 285 nm. If for any reason you need to drop a data point from your analysis, **justify it**. Explain why it may be erroneous and how its inclusion would affect your data.
- V. **Calculations.** Answer the questions at the end of the experiments. The starting equation as well as the steps involved in calculating a value are shown. Label the equation – e.g, Clausius-Clapeyron equation - and state what all of the variables mean. If the equation is based on any assumptions (*e.g.* an ideal solution), state those assumptions and explain why they should be valid for the present circumstances (or indicate that they may not be valid, and return to this issue in your discussion of error). All units and unit conversions are shown. Keep track of significant figures! *As inputting equations using document software is very difficult, example calculations can be handwritten and attached at the end of the lab report.*
- VI. **Discussion.** After you have done your calculations, you need to determine if your values make sense, and exactly what your data means in terms of the system being studied. **Remember that these experiments have been done before** – in some cases,

many times, and **so reported values exist in the published literature**. Use SciFinder to *locate published values*, and include these values in your discussion, comparing your calculated value to the published value. Be sure to include a citation to the published data (see ‘References’ below). State the percent difference. Is your calculated value close to that reported or way off? If the latter is true, you need to include a discussion of *possible sources of error*. Do not simply list sources of error. Explain how each source would lead to the observed error, or indicate that that source of error is inconsistent with your observations (and therefore probably less important than some other source).

For example, suppose water contamination in your sample would be expected to lead to a value of the heat capacity that is too high. You observe a value of the heat capacity that is lower than the literature value. In this case, water contamination is probably not the dominant error, and you need to find some other form of error that is responsible. In the report, you should mention the possibility of water contamination, point out that the expected error is inconsistent with the observed error, and then consider the real cause of the error. Perhaps your expectation with regard to how water would affect the heat capacity is wrong (suggest why this might be the case), or suggest an alternative error that would lead to too low a value of the heat capacity.

VII. **Conclusion.** Summarize the findings of your experiment, reporting the calculated values and their agreement with previously published results.

VIII. **References.** When comparing your data to literature values, you must always cite your source. One standard reference format for journal articles is, e.g.:

Jones, T.A.; Smith, M.S. and Brown, G.G. Ten ways to remove barnacles from steel hulls. *J Naval Tech* 34 (2010) 5-10.

Lab Grade

Lab grades will be based on satisfactory performance of the experiment, the quality of lab notebook keeping, the 7 laboratory reports, and the special project report and oral presentation. At the end of the semester, a numerical grade will be reported to the course lecturer for use in calculation of the final grade for the course. The lab grade typically accounts for ~30% of a student’s score, but the lecture instructor will explain his specific grading scheme.

Grading Scheme

Students will receive 30% credit for an experiment just for performing the experiment. This amount can be reduced if there is evidence of poor conduct in the laboratory, such as poor safety practices or nonparticipation. A student who misses an experiment without an excused absence will receive 0 credit for this component. Also, any evidence of academic dishonesty, such as fabricating data or plagiarizing a laboratory report, will lead to 0 credit for this component (*i.e.* A student is better off not turning in a lab report than turning in a fraudulent one).

The remaining 70% credit will be awarded based on the quality of the laboratory report. Guidelines are given in the “Lab Reports” section of this syllabus. Reports will be assigned letter grades that will be converted to percentage grades at the end of the semester according to the table at the right.

A	100
B	85
C	75
D	65
F	50

Note that assigning a score of 50% to the F grade essentially awards students 0 credit for the report. Also note that this scheme contains a high premium for A work. A level reports are hard to write, and are rewarded accordingly. The following criteria are used to assign letter grades

- A: The report clearly states the objective and how the method will lead to that objective. The reader is led from the raw data through data analysis to the conclusion. Relevant comparisons to literature values are made. A clear discussion of possible sources of error is included, which both identifies the error and indicates how it would affect the results.
- B: The report largely follows the pattern above, but is deficient in one aspect. Typical problems include a poor discussion of error or the lack of a clear connection between the analysis and the conclusions.
- C: The report includes the data and performs the necessary analysis, but does so without providing the reader any insight into the problem. The report does not discuss the analysis adequately nor does it make a clear connection between sources of error and the observed error.
- D: The report includes at least some of the relevant data and analysis, but contains serious problems. Incorrect or missing units and errors in data analysis are present, and the discussion of results and possible sources of error are irrelevant or erroneous.
- E: Even the most basic aspects of the data analysis are performed incorrectly.

Some general rules to help you write good reports:

Presenting data: Present data clearly. Indicate where the numbers came from (*i.e.* if you took multiple data varying a concentration or other parameter, indicate the value of the parameter), give units for the number, and use the correct significant figures. Figures and tables should include captions, and axes/columns should be clearly labeled. If for any reason you need to drop a datapoint from your analysis **justify it**. Explain why it may be erroneous and how its inclusion would affect your data.

Using equations: If you use an equation, explain where it came from (include a derivation if you like) and what all of the variables mean. If the equation is based on any assumptions (*e.g.* an ideal solution), state those assumptions and explain why they should be valid for the present circumstances (or indicate that they may not be valid, and return to this issue in your discussion of error).

Error analysis: Do not simply list sources of error. I repeat: **Do not simply list sources of error!** *Compare your results to literature values*, and discuss possible sources of error that could be responsible for the discrepancy. Explain how each source would lead to the observed error, or indicate that that source of error is inconsistent with your observations (and therefore probably less important than some other source). Keep it brief: this is not an excuse section.

For example, suppose water contamination in your sample would be expected to lead to a value of the heat capacity that is too high. You observe a value of the heat capacity that is lower than the literature value. In this case, water contamination is probably not the dominant error, and you need to find some other form of error that is responsible. In the report, you should mention the possibility of water contamination, point out that the expected error is inconsistent with the observed error, and then consider the real cause of the error. Perhaps your expectation with regard to how water would affect the heat capacity is wrong (suggest why this might be the case), or suggest an alternative error that would lead to too low a value of the heat capacity.

If no literature value is available, identify possible sources of error and explain how each would affect your results. You may also suggest ways in which the experiment could be improved, but if you do so, explain what error the improvement addresses and why you believe that error is significant. Generally, the only way to improve an experiment is to address the largest source of error, since eliminating small errors will not lead to significant improvements.

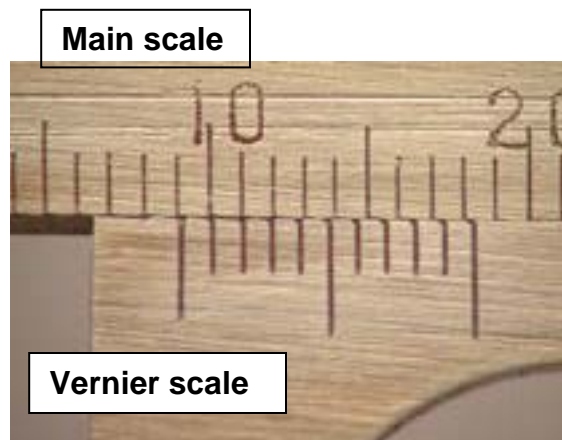
Citations: When comparing to literature values, always cite your source. Use an accredited citation format! By now, you should be familiar with a number of them. I don't care which you use, but pick one and use it correctly.

A Note on Using Vernier Scales

Various equipment in the physical chemistry laboratory, including the polarimeters and the barometer, incorporate a vernier scale to allow them to be read more accurately. The instructions below will tell you how to read a vernier scale.

(Taken from: <http://genchem.chem.wisc.edu/labdocs/modules/scales/scalesvernier.htm>)

A Vernier scale is an auxiliary sliding scale that can be used to more accurately read the values on a fixed main scale. Its purpose is to allow accurate readings, rather than estimations, between the smallest graduations on the fixed scale. A vernier scale commonly has ten graduation marks. Each division on the Vernier scale is nine-tenths of the size of the finest division on the main scale.



To use the Vernier scale, read the main scale to the last certain digit. The last certain digit on the main scale is the graduation just below the zero on the vernier scale. The mark on the vernier scale that directly lines up with a graduation mark on the main scale is the last digit in your reading.

The measurement shown in the image above is **9.2 mm**. Since the zero mark of the lower Vernier scale is past the 9 mm mark and the two mark on the Vernier scale lines up exactly with a graduation mark on the main scale.

Safety Warning:

Any student who has a sensitivity toward chemicals or who may be pregnant or becomes pregnant during the course is strongly advised to check with his or her doctor to determine if taking this course may pose a hazard to his or her health. A list of chemicals to be used in the laboratory experiments will be made available upon request.

There is strong evidence that a mother's exposure to volatile solvents during pregnancy can lead to birth defects (See: <http://www.cnn.com/HEALTH/9903/23/pregnancy.exposure/>). It is recommended that pregnant students do not take the laboratory component of the course. Please speak to the course lecturer about the risks and the means by which the department can accommodate you.