CHEMISTRY 4610
PHYSICAL CHEMISTRY LABORATORY

Syllabus Fall 2017

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Office Hours: Tues 10:00 AM – 12:00 PM; Thurs. 3:00 – 5:00 PM.

Required Text None. Printed handouts given.
Required Material bound composition lab notebook

Course Structure: The physical chemistry laboratory will meet once a week, on Tuesdays from 2:00 – 5:50 PM in 439 New Ingersoll. Each lab session will consist of roughly 30 minutes of introductory lecture, and then students will go into the lab to work. Introductory material will mostly concern the theory behind the lab and the finer points of lab 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 be working in small assigned groups of 2*.

Experiments
There are 7 experiments, but due to limited equipment, different groups will be working on different experiments each week: consult the experiment schedule handout for your schedule. During weeks that a group is not working in the lab, each student will be working on a special project (to be determined), for which each student will give a 10-15 minute oral presentation during the last lab meeting. Note that for the second lab meeting, all students will be working on the NMR experiment: lab tech, Isanna Agrest will show the working of the NMR instrument and be collecting data.
Week 1: Handout: Introduction to the Scientific Literature
Week 2: NMR Determination of Keto-Enol Equilibrium Constants/approval of journal paper
Weeks 3 – 9:

- Vapor Pressure of a Pure Liquid
- Differential Scanning Calorimetry
- Binary Solid-Liquid Phase Diagram
- Surface Tension of Solutions: tensiometer and slide method
- Heats of Combustion
- Inversion of Sucrose

Weeks 10-13: All students working on a special project
Week 14: Oral presentation (Power Point)

Modifications for Specific Experiments:
NB: You are not required to report some of the calculations given in the handouts as outlined below.

NMR Determination of Keto-Enol Equilibrium Constants
Needed:

- Spectra, and a brief discussion of the assignment of peaks
- Correct calculation of the equilibrium constant and a discussion of sources of error
- Calculation of ΔG for the reaction

<u>Not needed:</u>

Do not integrate over every peak assigned to a given molecule; choose one peak for each tautomer, integrate each, and analyze accordingly

Vapor Pressure of a Pure Liquid

<u>Needed:</u>

Discussion of systematic and statistical error, including error bars on fitted parameters (see "Analysis and Presentation of Data" appendix in Lab Manual),

Assessment of the T-dependence of ΔH and Z, and how each affects the accuracy of the calculated results.

Correct propagation of error as appropriate

<u>Not needed:</u>

Do not make a "mercury correction" to external pressure

Do not attempt to calculate the entropy of vaporization

Do not use the method of "limiting slopes" to evaluate your error

Binary Solid-Liquid Phase Diagram

<u>Needed:</u>

Estimated value of the eutectic point

Careful discussion of error

<u>Not needed</u>:

Actual eutectic run (do this if time permits)

Formal lab report – data analysis only

Surface Tension of Solutions

<u>Needed:</u>

Discussion of the scatter of observed heights (sensitivity to cleanliness of glass/slide method)

Average and standard deviation associated with each point

You can drop data points from the analysis, provided you present the raw data and justify your decision to drop the point.

<u>Not needed:</u>

Drop some runs if you are short of time

Inversion of Sucrose: data analysis only; no formal lab report

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<u>Lab Report Due Dates</u>

The literature report is due **Sep. 5th** (2nd class). Lab report due dates are given on the lab schedule.

NB: Late reports will be docked one full letter grade for each week late. Reports must be in hardcopy – e-mailed lab reports will not be accepted.

<u>Format of Laboratory Reports</u>

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 (See Appendix I in lab manual). 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, detailed calculations must 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 Scholar (available on Brooklyn College library website under "all databases" tab) 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 ('way off' > 15%)? 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 8 laboratory reports, and the 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.

<u>Grading Scheme</u>

Students will receive 50% 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 50% 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

<u>A</u>: 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.
<u>B</u>: 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.
<u>C</u>: 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.
<u>D</u>: 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.
<u>F</u>: Even the most basic aspects of the data analysis are performed incorrectly.

Students are given the option of resubmitting lab reports where the objective of the experiment has been misunderstood or missed. This procedure ensures that the student learns and understands the objective of the experiment; otherwise, the opportunity to learn is lost.

One lab grade credit will be awarded for the **keeping of the lab notebook**. 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 and adjustments to the awarded credit will be made depending on the quality of the notebook.

<u>Safety Warning</u>

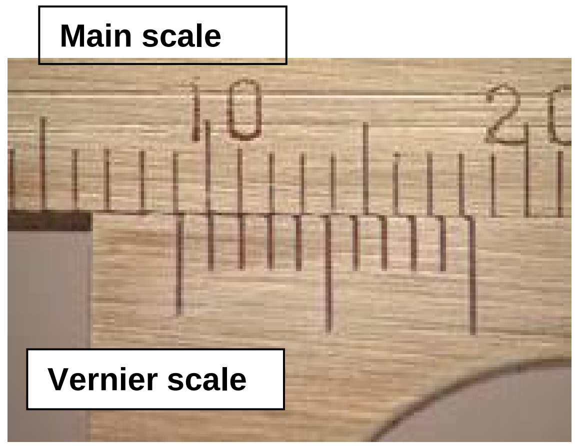
Any student who has a sensitivity toward chemicals or who may be pregnant or becomes pregnant during the course is strongly advised to check with their doctor to determine if taking this course may pose a hazard to the student's health or that of the unborn child. A list of chemicals to be used in the 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. 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.

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.