CHEMISTRY 4610
PHYSICAL CHEMISTRY LABORATORY

Syllabus Fall 2020 - Online

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Office Hours: Weds 1:00 –3:00 PM. On Blackboard Collaborate

Required Text None. PDF of experimental procedure and data analysis is posted on the Blackboard website for the class under Course Documents.

Course Structure: Due to the restrictions on in-person interactions required for the safety of all during the COVID-19 pandemic, completing laboratory experiments in the lab is not possible. Therefore, virtual lab tutorials have been created. These consist of voice-annotated power points that include links to appropriate videos on procedure and concept available online. Student are to view and understand these tutorial materials, which are posted on the chem 4610 Blackboard website under the Course Documents tab. Separate folders have been created for each experiment. The lab manual---used for in-class experiments---is also found under Course Documents. It contains printed procedures for each experiment, the theory, and the equations needed for data analysis. The data itself for each experiment is provided since you obviously are not able to collect your own. Students are to analyze the data according to the instructions for calculations given in the experiment write-up. General instructions for writing lab reports are given below, and more extensive instructions are included in the lab manual PDF, also posted on Blackboard. ***Modifications for specific experiments are given below.*** Each lab report, including the scientific literature report, is submitted the week following the assigned experiment. We will arbitrarily set the assignment day as Friday, since this is when the lab traditionally meets. Therefore, lab reports are due to be uploaded to the Blackboard *Assignments* folder for that experiment by Friday at 5:00 PM. (NB: You will *NOT* be submitting your reports to the OneDrive folders as previously announced). Only single documents are to be submitted (more on this below). The first 'experiment' is the literature research assignment (see lab manual), which is due on Friday, September 4. At 5:00 PM

Experiments
There is one literature research-based assignment and 7 lab technique-based experiments that are listed below. During the remainder of the semester, students will carry out a computational experiment using the freeware program GAMESS, available on the website, https://chemcompute.org/gamess. Students should first acquaint themselves with the "How to Perform Quantum Calculations" under the GAMESS Experiments → General Instructions tab, utilizing both the Background Theory and Lab Experiment. Next, under the Physical Chemistry tab, choose the experiment:

- **Transition State Theory: Kinetics of Ammonia Production**
  Reference: KM Stocker 2018
  Background Theory / Prelab / Lab Experiment

This is the computational experiment to be performed. Please fill out the pre-experiment survey. Following the calculations for the thermodynamic parameters for all molecular species involved in the reaction steps, complete the calculations, analyses, and answer all the questions outlined in the Results and Discussion section. This will be submitted for grading along with the output files for the 4 transition states associated with the reaction process. You should also complete the post-experiment survey. As this project will be very new to most students, five weeks of class time are devoted to it.

The order of the experiments is as follows (all due at 5 PM on the due date):

Introduction to the Scientific Literature – *due Sept. 4*
NMR Determination of Keto-Enol Equilibrium Constants– *due Sept.11*
Vapor Pressure of a Pure Liquid– *due Sept. 25*
Differential Scanning Calorimetry– *due Oct. 2*
Binary Solid-Liquid Phase Diagram – *due Oct. 9*
Surface Tension of Solutions: tensiometer or slide method– *due Oct. 16*
Heats of Combustion– *due Oct.23*
Inversion of Sucrose– *due Oct. 30*
GAMESS computational project– *due Dec. 4*

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 (raw data), and a brief discussion of the assignment of peaks. Peak assignment for protons of each type for each tautomer must be made using integrated peak areas (given) and the known relative abundance of each type of proton in each tautomer. Show calculations.
Correct calculation of the equilibrium constant and a discussion of sources of error
Calculation of ΔG for the reaction

Vapor Pressure of a Pure Liquid
Needed:

Do Calculations (p 27) and answer questions in first paragraph of Discussion (p 27)

Binary Solid-Liquid Phase Diagram
Needed:

Comparison of heat of fusion value obtained for naphthalene using DSC vs. value calculated in this experiment. How do both values compare to a published value?
Estimated value of the eutectic point
Careful discussion of error

Not needed:

Formal lab report – data analysis only

Surface Tension of Solutions
Needed:

Discussion of the scatter of observed 'weights' (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.

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

Format of Laboratory Reports

Laboratory reports will consist of the following parts:

I. **Title of the experiment**, the name of the student

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 cumbersome, a detailed representative calculation must be handwritten and attached at the end of the lab report, showing unit analysis (i.e, if the same calculation is used repeatedly, you only need to show all steps once). Handwritten pages may be scanned or photographed (screen shots) and embedded in a Word document (make sure it is legible). ONLY A SINGLE DOCUMENT WILL BE ACCEPTED FOR EACH LAB REPORT. MAKE SURE YOU INCLUDE YOUR NAME IN THE FILENAME (Half a point will be deducted for not attending to this detail. I cannot keep track of 10 files called 'experiment 5.doc.')

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*. *Consider that you may not have done the calculation correctly;* the data is actually fine, you just made errors in calculation. *Therefore, repeat the calculation*! Unit analysis often shows where the error is made.

VII. 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.

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

IX. **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

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 counts as 25.3% towards you final numerical grade. All experiments except the computational project are weighted as 10 points; the computational project is weighted 40 points.

NB: Reports submitted up to one week late will be docked 1 point out of a possible 10; up to two weeks late the deduction is 2.5 points. Reports will not be accepted later than two weeks past the due date.

Grading Scheme

As everyone will be working with the same data set, data forgery is not an issue. However, duplication of calculation pages and plagiarizing of laboratory reports is. Evidence thereof will result in no credit for the lab report for all participating parties.

The following criteria are used in numerical grading:

10 pts: 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.

8.5 pts: 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.

7.0 pts: 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.

6.5 pts: 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.

5 pts: Even the most basic aspects of the data analysis are performed incorrectly.

Students may be 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.