Physical Chemistry Laboratory Class
(Chem 342L)
C 108
Fall 2007

Department of Chemistry
South Dakota School of Mines and Technology
subject to change

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Physical Chemistry Laboratory Class
Schedule of Labs
Each lab will have a pre-lab lecture. This will signify the opening of a ‘window’ of time in which the lab can be performed. The due date of each lab will signify the close of the window and following that date that lab cannot be performed without instructor permission. Labs will be posted on WebCT for you to print out. Dates are subject to change!

<table>
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<tr>
<th>Lab #</th>
<th>Experiment</th>
<th>Pre-lab Date</th>
<th>Due Date</th>
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<td>Check In and Lab Video Thermometer Calibration</td>
<td>Sept. 13\textsuperscript{th}</td>
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<td>2</td>
<td>1 Adiabatic Gas Behavior</td>
<td>Sept. 13\textsuperscript{th}</td>
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<td>2</td>
<td>Determination of $\Delta_{\text{fus}}\text{H}$ Using Freezing Point Depression and Construction of Liquid-Solid Phase Diagram</td>
<td>Sept. 20\textsuperscript{th}</td>
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<td>3</td>
<td>Heat of Reaction of Magnesium and Hydrochloric Acid*</td>
<td>Oct. 4\textsuperscript{th}</td>
<td>Oct. 25\textsuperscript{th}</td>
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<tr>
<td>4</td>
<td>Kinetics of Crystal Violet</td>
<td>Oct. 18\textsuperscript{th}</td>
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<td>Binary Liquid-Vapor Phase Diagram</td>
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<td>Lab Books Due</td>
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<td>7</td>
<td>Viscosity of Liquids Part I: Low Viscosities*</td>
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* Hard copies of the lab will be handed out.
Lab Grades:
Your grades will come from four sources:

**Memos:** The individual grades you receive on your memos for each experiment. Each memo will be graded out of 10 points. Memos will be posted by each group to WebCT. A detailed explanation of what is expected in each is found in the Lab Reports and Memos section. Late memos will be given a grade of zero.

**Lab Books:** Each group must keep a detailed lab book worth 100 points. These must be detailed enough that someone else in the lab could duplicate your experiment. Variations from the described method, conditions, time, etc. must be listed. These will be turned in at on November 29th and keep in your instructors office to be checked out as needed for your final reports. Points will be deducted for poor technique in your lab book (no dates, signatures, etc.).

**Discretionary:** There will be an additional 200 discretionary points for the course. You will start with these points and they may be taken away at any time for performance or behavior that may warrant it, including improper use of equipment. Failure to provide quality results to the class will result in a large deduction from these points. Peer evaluations will also result in loses of points for failure to work well with your lab partner.

**Final Report:** Each student will be assigned one experiment in which they will develop an extensive report worth 400 points. You will use the results for the experiment posted from each group to develop a data pool to use for this. You will have to choose which data is reliable and which is not. To do this you will use statistics and information from their lab books or memos that you feel is important. This report is to be approached as if you were submitting to an ACS journal (Journal of Physical Chemistry A, B, or C). A detailed explanation is found in the Lab Reports and Memos section.

Reports are due December 13th, with 100 point deductions for each day late. No lab reports will be accepted following December 17th.

The following grading scale will be used as a starting point. The grade cut offs may be lowered, but will not be raised from those listed below:

A: 90%     B: 82%     C: 74%     D: 66%     F: <66%

Lab Guidelines:

Students will work with a partner throughout the length of the lab. You will get to pick your partner, as you will need to work with someone that you can meet with outside of the lab. This will make calculations and other things easier.

Equipment for this lab must be handled with the utmost care. In some cases we only have one instrument and if it breaks it means problems. For this reason if you have any questions about how to operate an instrument, balance, IR, etc., please ask before making assumption.
Students will not be required to be in the lab when they are not working on an experiment. It is understood that you may want to work on calculations and writing outside of the lab. It will be your responsibility to schedule a time to perform all labs during the allotted lab time. Scheduling of lab times will be done following the pre-labs.

**Safety Rules:**

As there may be multiple labs going on at the same time it will be very important to be aware of yourself and others in the lab.

- You will not be allowed to do an experiment without proper preparation in advance. If you are not prepared to perform a given lab you may be asked to leave until you are. These situations may arise from one of the following:
  - If you did not read the lab draft in advance and show that you understand important points.
  - If you missed a pre-lab lecture for a lab. You will need to meet with the instructor or TA in this case.

- You must wear proper attire; this includes department approved safety goggles. Failure to do so may result in you being asked to leave the lab for that day.

- Any unauthorized experiment at any time will result in the immediate assignment of a final grade of “F” for the course.

- Dispose of laboratory materials in the proper waste bottles located in the hood or designated area. With multiple labs taking place at the same time there may be multiple waste bottles available, so make sure you use the right container. If you are unsure of how to dispose of something **ASK**!

- Laboratory drawer replacement items are free-of-charge only on the first scheduled meeting. After the initial checkout day, you will be responsible for the replacement of broken or missing items. This includes, but is not limited to, items that are lost due to your failure to return them to your drawer or your failure to lock your drawer. Replacement items are to be paid for at the time of their acquisition.

- Unexpected events (emergencies, spills, accidents, etc.) must be brought to the immediate attention of your TA. Do not leave the lab without informing your TA of the event.

- The use of tablet/laptop PCs in the lab will be prohibited. You may store them out of harms way during the lab, but you may not use them in the lab.

**Fine for Failing to Check-in or Return Key:** All students who have checked out a lab locker and drawer key are required to check the locker and key in at the end of the semester or earlier if withdrawing from the course. A fine of $30.00 is assessed for failure to check-in and a fine of $100.00 is assessed if you lose your key or fail to turn it in upon check-in. If circumstances force you to withdraw from the lab before the end of the semester, you should make arrangements with the storeroom manager (Kris Grinnell, C 101) to check in your desk and key in order to avoid the fines.

**ADA Statement**

At the recommendation of the ADA Advisory Committee, we are asking that you use the following language as the ADA statement:
Students with special needs or requiring special accommodations should contact the instructor, (Justin Meyer, at 394-2431) and/or the campus ADA coordinator, Jolie McCoy, at 394-1924 at the earliest opportunity.

**Freedom in Learning Statement**

The following statement should be used on all syllabi.

*Freedom in learning.* Students are responsible for learning the content of any course of study in which they are enrolled. Under Board of Regents and University policy, student academic performance shall be evaluated solely on an academic basis and students should be free to take reasoned exception to the data or views offered in any course of study. Students who believe that an academic evaluation is unrelated to academic standards but is related instead to judgment of their personal opinion or conduct should contact the dean of the college which offers the class to initiate a review of the evaluation.
Physical Chemistry Laboratory

Lab Reports/Memos

Part of this class consists of an introduction to technical writing. The laboratory report is therefore one of the most important components of this class, and will constitute the bulk of your grade. Attention to the following details is important:

- Lab reports/memos should be submitted to WebCT via a PDF file. To do this you can use a variety of programs to convert to a PDF file (CutePDF) or use adobe writer if you have it.

- You must provide access to your raw or handwritten data. A reference to your lab book is sufficient for this. A copy of your raw (handwritten) data must be included at the end of the lab report. If your raw data consists of multiple data runs that have hundreds of points, an example graph/table can be used with a description that notes that there are numerous other ‘similar’ data sets.

- **EACH STUDENT IS EXPECTED TO PREPARE HIS OR HER OWN LABORATORY REPORT**, even if you had a lab partner for the collection of the experimental data. You are expected to submit a lab report in your own words.

**Formatting:** All reports/memos must be double spaced, with left and right margins of 1 ½ inches. Between sections there should be four spaces (extra carriage return). This formatting will allow clearer comments to be written by the corrector.

**A) Memo Organization (1-3 pages of text)**

A lab memo will consist of the following sections:

1) Abstract
2) Experimental Procedure
3) Results (Data and Computed results)
4) Discussion/Conclusion

What you are expected to include in each section:

1) **Abstract:** This should be a short description of what was done and the results. A person reading this section should be able to tell if this ‘report’ is something they want to reference, either from the importance of the experiment, or the results stated.

2) **Experimental Procedure:** This is not a recitation of the lab handout. Instead a reference to the lab handout followed by any notable changes or deviation that were performed. A good guideline is to give enough information that someone could duplicate the experiment who has not seen the lab before and they could get the same results. Remember to explain how you can to your results, calculations, etc.

3) **Results:** This section will give your calculated or determined results. Tables and graphs should be labeled and referenced. You should not have a lot of text in this section, but it needs to be coherent, and flow. If you have a large amount of data you can use some summary graphs and include the rest of your data in an appendix.

4) **Discussion/Conclusion:** This is where you should discuss the outcome of the experiment. It should also have any observations you made during the lab. You should
also answer any questions that are in the lab write up in this section. You may keep this fairly short and to the point.

Aside from the memo, you are expected to post your data in a separate file (excel or text) to WebCT so everyone can access it. Failure to do so will result in a deduction of discretionary points.

B) Report Organization
A lab report will consist of the following sections:
Those found in a typical ACS Journal:
1) Abstract
2) Introduction
3) Experimental Procedure/Methods
4) Results (Data and Computed results)
5) Discussion
6) Conclusion/Concluding Remarks
7) References
Additional information to be attached in separate file:
1) Sample Calculation(s)
2) Sample Error Calculation

Each section is described in detail:
1) Abstract: The abstract is a short (1-paragraph) summary of the experiment and the results. An example of a good abstract is:

   Potentiometric measurements were used to 1) calibrate a copper-constantan thermocouple at various fixed points and 2) to determine resistance values of “unknown” resistors in an electrical circuit. The calibration curve for the thermocouple was similar to one drawn using values found in literature. The resistance values were found to be within the expected or related tolerances. The one thing to use some judgment on is to include your results (values) with error in the abstract. This should be done if you have one or two targeted values, but if you have numerous, then you will make the abstract too long, and they should be referred to as ‘values’ as in the example.

2) Introduction: This should be a short description of the theory and reasoning for the lab. You should also reference other work in this field. You should not talk about the procedure or conclusion here, just the properties being measured, properties being calculated, and possibly the interest of the properties to daily phenomenon. This is where you will state a lot of the important equations used in the experiment. These equations should be labeled, and if needed, referenced. An example:

   \[ \text{HCCO} + \text{NO} \rightarrow \text{Products} \]  

   Where the citation is linked to a reference in the back of the report. By labeling your equations you can reference to them easily in the results or discussion section.
3) **Experimental Procedure:** This is not a duplication of the lab handout. Instead a reference to the lab handout followed all notable changes or deviation that were performed. You may also reference any other literature methods that are similar to those performed. A good guideline is to give enough information that someone could duplicate the experiment who has not seen the lab before and they could get the same results.

3) **Results:** This section will give your calculated or determined results. Tables and graphs should be labeled with captions and referenced. You should not have a lot of text in this section, but this is part of a paper, and therefore you need to have text that links all tables and graphs together. You should use this text to say how you came about the results, for example:

By using equation 1 followed by equation 2, a value of pressure was determined, as shown in table 1.

If you have multiple graphs or tables of the same type you should include one here and reference the rest in an appendix. Remember, any physical quantity, whether raw data or calculated, must have an uncertainty associated with it.

4) **Discussion:** This is often the most difficult but also the most important part of the report. Here, you are expected to demonstrate an understanding of the experiment, the principles on which it is based, and the physical significance of the results. This will often require extra reading in good reference books. The point is that you must think about what the experiment really means; it usually has some significance beyond a mere collection of data values.

   Included in this section should be a qualitative discussion of sources of error in the experiment. Discuss the assumptions that are made in the data analysis and describe ways in which one might improve upon the experiment or the analysis.

   The discussion section should include general interpretation of the data as well as physical interpretation of the results. The point here is to convince the grader that you understand what is going on, and are not just following a recipe. For example, in a spectroscopy experiment, identification of the spectral lines is important and the spectroscopic constant should be interpreted in terms of molecular structure and motions.

   Some of the lab handouts pose questions about the meaning of the experiment, etc. These questions should be answered somewhere in the discussion section. But do try to answer these questions in the context of the report, and not just list off answers.

6) **Conclusions/Concluding Remarks:** This is often a short summary of the implications of the results, and the future of the work. This could be what would be changed if the work is duplicated for better results, or what chemicals would you test for in the future.

7) **References:** If at any point in the report you refer to any books, journal articles, etc., they should be referenced at the end of the report. Use the citation format followed by the Journal of Physical Chemistry (ACS format). For example:


See an ACS Journal for more examples of the format.
Other Things Included in Separate File

1) Sample Calculation(s): Typically, your raw data may require some calculations in order to obtain the final result. Write down all non-trivial steps for one complete sample calculation. Even if you used a computer, you still must include this for one data point in order to show that you know how to perform the calculations. If you needed to derive an equation, the derivation should be in the report, just as if this were an exam or homework problem in a lecture course. If you used an equation from the handout or book, journal article, etc., be sure to reference the source.

After performing your calculation, ask yourself whether they make sense. If your answer differs from a literature value by 25%, the discrepancy may be due to experimental error. If the discrepancy is a factor of 1000, the problem is more likely in your calculations.

2) Sample Error Calculation: Your results section must include uncertainties in all calculated quantities. In the error analysis section, show how these uncertainties were calculated. This must be done by the propagation of errors technique; see the section entitled “propagation of errors” or Shoemaker et. al., chapter 2 for details. Note that qualitative discussion of errors should be in the discussion section, not here.

A common mistake is to quote an error bar by comparison with a literature value, i.e., to state that

\[ \% \text{error} = \frac{\text{(yours}-\text{theirs})}{\text{(theirs)}} \]

This is not adequate in most cases. The point of the propagation of errors technique is to start with an educated estimate in the uncertainty of the raw measurement, and convert this into an uncertainty in the final calculated answer. In this way, one obtains an intelligent estimate of the uncertainty in a result, even in the absence of any literature values.

C) Writing Style

A well-written technical report uses a writing style that differs slightly from that used in fictional literature, popular writing, etc. Scientific reports should be written in a moderately formal style that emphasizes clarity. In general, the use of the first person (“I”, “We”) should be minimized or avoided. Long, complex sentences should usually be avoided, although it is a good idea to vary sentence length to avoid putting the reader to sleep.

The choice of tense in technical writing may seem unimportant, but if used properly, verb tense provides subtle clues to the reader, which aid understanding. In general, reporting previously known scientific results or data should be done in the present tense, while reporting your own results should use the past tense. For example, when reporting generally accepted properties, you use the present tense:

Sulfur is a yellow crystalline solid that melts at 112°C to form a brown, viscous liquid.

If you are reporting your own observations, however, you use the past tense:
A 10-g sample of sulfur was heated in a melting-point apparatus and found to melt at 112°C. On melting a brown, viscous liquid was formed.

The most important aspect of technical writing is the conveyance of information. Assume that the reader has a reasonable knowledge of physical chemistry, but has never seen the lab handouts. Then ask yourself whether your lab report would be understandable to such a reader. If any section is vague or obscure, because the reader “just had to be there”, then your report needs revision.

Lab reports must be written in something that is reasonably close to standard, grammatically correct English. If you would be embarrassed turning your report in to an English professor, you should be embarrassed turning it in for a chemistry course. Occasional typing errors will be forgiven, but points will be taken off for sloppy writing habits such as incomplete sentences, repeating instances of poor grammar, spelling, etc. In severe cases of poor writing, lab reports may be handed back to you for revision.

*If you have any questions about your final report format, or what should be in it, please schedule a time to sit down with your instructor.*