Chemistry 311L-312L
Physical Chemistry Laboratory
1998-99

General:

This is a laboratory course designed to accompany Chemistry 311-312. It meets Tuesday afternoons and carries one credit for each semester. The text is David P. Shoemaker, Carl W. Garland, and Joseph W. Nibler, *Experiments in Physical Chemistry*, 6th ed., McGraw-Hill Book Co., New York, 1996.

Experiments:

There will be seven experiments each semester. Except for the first experiment, the experiments will be taken from the textbook. Instructions for the first experiment will be handed out in class. For some experiments, our apparatus differs from that in the textbook. In this case, an additional instruction sheet will be handed out by the instructor.

For each experiment, it will be necessary to (1) set up apparatus; (2) carry out the experiment and take data; (3) make calculations and draw conclusions; (4) write a report. There will be two laboratory periods for each experiment. If you have read and understood the instructions for the experiment before coming to class, the data for nearly every experiment can be taken in the first period, leaving the second period for calculations and report writing.

Students will work in pairs for all experiments. Lab partners should work together in setting up apparatus and taking data, but each student should record all data in his or her notebook, and should work individually in performing calculations and writing reports.

Reports:

A written report must be turned in for each experiment. The report will be due at the beginning of the period in which the next experiment is begun. The report for each experiment should be written in the same format as an article in *The Journal of Physical Chemistry* and must be typed and double spaced. The laboratory report may be turned in electronically to the instructor's folder on the college's academic volume instead of being turned in on paper. Use WordPerfect, Microsoft Word, or Claris Works for the Macintosh. A copy of the notebook pages for each experiment must be turned in as an appendix to the report, or turned in separately if you submit your report electronically.

Grading:

The grade for the course will be determined by the grades on the reports. The grade on each report will be determined as follows:

notebook	10 points
calculations and accuracy	20 points
error analysis	10 points
report format, organization, etc.	10 points
total	50 points

There will be a penalty of 10% (5 points) for late reports, with possibly a larger penalty for very late reports.

Comments

General:

Chapters I, II, III, and XVI through XXII of the text contain information that is useful in setting up apparatus, making calculations, and writing reports. These chapters are probably even more useful than Chapters IV through XV, which contain the instructions for the experiments.

The laboratory notebook:

In a research laboratory, the notebooks of individual researchers are legal documents that can be used to establish priority for discoveries, eligibility for patents, etc. There are instances of failures to obtain patents because notebooks were not kept properly. One objective of this course is to help you learn laboratory work habits that would acceptable in a research laboratory, including proper notebook keeping.

The physical chemistry notebook must be a bound notebook, so that it can be seen whether any pages have been removed. You must enter the date and the name of the experiment on every page of the notebook, and the pages must be numbered. At the first of the record of any experiment, you should give a reference to the laboratory textbook and/or any other instructions you have received.

All notebook entries must be in ink, and must be recorded at the time the work is done. It is absolutely not acceptable to record something on a paper towel, on a loose sheet of paper, or in another notebook, and then to copy it into the notebook. If you make a mistake and write down an incorrect value or other mistake, do not obliterate the mistake. Draw a single line through it so that it can still be read and then record the correct value. If data are taken in such a way that only one partner can make a record while the data are taken, the other partner is permitted to copy the data into his or her notebook, but must do so as soon as possible.

The notebook record must be complete. A narrative record of everything you do must be included. For example, if you make up a solution with a solid solute, you should write a sentence saying what solution you are making. You should then record the weighing data, including a statement of what balance is used, what the source of the solute was (manufacturer's name, lot number, etc., if you have the manufacturer's bottle). If you weigh by difference, the tare weight and the gross weight should be recorded in the same column and the difference written beneath them. You would then write a sentence describing the transfer of the solute to a volumetric flask and state what solvent you use (deionized water, reagent grade ethanol, etc.). If you make any measurement with an instrument, you should record the name, model number, and location of the instrument used, as well as all instrument settings. If the instrument produces charts, you should number the charts and put the date on them, and should refer to them in your notebook by number.

If you and your partner divide up some tasks, record in your notebook what it is that your partner has done, and give a reference to his or her notebook by page number. If you use a solution prepared by Mr. Goode or one of his assistants, record that fact and record everything that is on the label of the bottle.

Make your notebook so that it can easily be read by someone else and so that a reader would know exactly what you did and how you did it. If you have data that can be

organized in table form, it is more easily read in that form. You should also construct your notebook so that you could repeat the experiment in exactly the same way a year later by referring only to your notebook.

Calculations:

All physical chemistry experiments require some numerical calculations. Some students prefer to record their calculations in their notebooks, but this is not required. However, if you record the calculations on loose sheets of paper, each sheet should be dated and numbered (and saved). Some experiments require use of a computer program, a spreadsheet, or graphing software. You should keep a record of what you do as you do it. For example, if you make several graphs using CricketGraph, you would number and date the graphs and would write a separate record of what function you graphed in each one, etc. in your notebook. Do everything in an organized way. If you have to come back later and repeat a calculation, it will be easier if you don't have to search around trying figure out what you did the first time.

Error analysis:

Every numerical value which you report must be accompanied by an expected error. It is customary in physical chemistry to report expected errors at the 95% confidence level. For example, if we report a value of the enthalpy change of vaporization of water at room temperature to be 43.5 ± 0.6 kJ mol^{-1}, we mean to communicate our opinion that the interval from 42.9 kJ mol^{-1} to 44.1 kJ mol^{-1} has a 95% probability of including the correct value.

You must first estimate the errors in any quantities which you measure directly, and must then propagate these errors through your calculations if you calculate other quantities from the measured quantities. Carry out the error propagation calculations as you carry out your main calculations.

In the physical chemistry laboratory you will measure quantities that have been measured and reported in the literature. Therefore, you can determine the difference between your value and the accepted value for most quantities that you measure. Most currently accepted values have expected errors which are much smaller than your expected errors, so you can call the difference between your value and the accepted value your "actual error." Compare your expected error and your actual error to see how realistic your error analysis is. Give the expected error and the actual error in the same units. That is, do not report the expected error as 0.6 kJ mol^{-1} and the actual error as 1.2%.

Insignificant digits should not be reported. It does not make sense to report that an enthalpy change of vaporization of is 43.521 ± 0.606 kJ mol^{-1}. An expected error needs only one digit (two digits if the first digit is 1). In the enthalpy change of vaporization given, although the 5 in 43.5 is not quite a significant digit, we include it, since the error is smaller than 1 kJ mol^{-1}. Just don't include anything that makes no difference or anything that doesn't make sense.

The report:

You can probably get the proper amount of detail in your reports by imagining that you are writing it for a reader who took a physical chemistry course last year, but who did not do the particular experiment. Use the format of research articles in the *Journal of Physical Chemistry*. There is a sample report in the laboratory textbook, beginning on page 15. The following things should be included in your report:

1. A title page. Include all of the obvious things. Also include in a lower corner the following double-spaced list for the instructor to use in reporting your grade:

notebook	____ of 10
calculations and accuracy	____ of 20
error analysis	____ of 10
report format, organization, etc.	____ of 10
total	____ of 50

2. An abstract.

3. A brief introduction that includes a statement of what is being measured and what is being calculated from the measured quantities, including the theoretical basis for the calculations. A reference to the physical chemistry lecture text or other sources should be included.

4. A brief description of the procedure. This does not need to include all the details, but should specify the general method and what kinds of instruments were used. There should be a reference to the laboratory textbook pages on which the details of the procedure are found. If the procedure is different from that in the textbook, this section might contain two paragraphs instead of one.

5. A description of the measured results and the calculations based on the results. Do not put every step of the calculation in the report, but tell how the calculations were done. Be guided by the practice in articles in research journals. Details of the calculation can be put in an appendix.

6. Your error analysis. You should include a list of possible sources of error, both systematic and random, with a statement of which you think are negligible. Your error analysis should lead to an expected error in each measured or calculated quantity.

7. A section in which the final results and errors are collected together. If several cases are considered, the results should be presented in table form. Each result should be accompanied by its expected error and if possible, the actual error.

8. Conclusions and/or discussion of your results. For example, if you are asked to test a model theory or formula, state whether the model or formula is invalidated by your results.

9. A legible photocopy or carbon copy of the notebook pages for the experiment must be included as an appendix to the report. Sample calculations, computer programs and output, instrument charts, etc., can also be included as appendixes.