

Physical Chemistry Laboratory I
CHEM 445
02F

There is no textbook for this laboratory course. Introductory material and detailed experimental procedures are available on the CHEM 445 Home Page (where you are now) as downloadable PDF files, using Adobe Acrobat Reader (available on the UD Website, <http://www.udel.edu/sw/>, under "Connectivity, Virus Protection, and Web Software"; also available as a link to Adobe on the CHEM 443 or CHEM 444 Home Page).

The following laboratory experiments will be done this semester. Because of limitations of equipment, the experiments will be done in rotation. Each student will do six experiments during the semester. All six experiments will be done with a lab partner. Schedules of the sequence of the experiments for each lab section will be posted on the laboratory door, 253E BRL, and are shown on the CHEM 445 Home Page.

Experiment 1:	Freezing Point Depression of Electrolytes
Experiment 2:	Partial Molar Volume of an Electrolyte
Experiment 3:	Effect of Ionic Strength on the Solubility of CaSO_4
Experiment 4:	Heat of Combustion of Naphthalene
Experiment 5:	Ratio of Heat Capacities of Gases
Experiment 6:	Vapor Pressure of a Pure Liquid

You should read the material for each experiment thoroughly **before** you come to the lab to do the experiment. A copy of the detailed procedure for each experiment will be available in a notebook at each station in the laboratory. You are not expected to memorize the steps in the experiment.

Because all of the experiments are being done simultaneously in each laboratory period, the Laboratory Instructor cannot describe each experiment at the beginning of each lab period. The Laboratory Instructor will provide help for you during the laboratory period. You should always check with the Laboratory Instructor before using the gas cylinders and when you are uncertain about using the equipment. You and your lab partner should discuss the experiments before you begin.

NOTEBOOKS:

Recording and documenting data is essential to research. It is better to record more information than less. All data are to be recorded in a bound research notebook with numbered pages and provisions for a carbon copy of each page. The Bookstore has acceptable notebooks: some with carbon paper and some without. Other places have equivalent notebooks.

Begin your notebook with a Table of Contents. Each experiment should be listed in the Table of Contents with a page number for the beginning of each experiment. Add to the Table of

Contents as you write each experiment. Put your name and address on your notebook – just in case you leave it in the lab.

Write a brief introduction to the experiment (purpose and general procedure) in your lab notebook **before** coming to the lab. The introduction should not be as detailed as the introduction in the written material for experiment. Do not copy directly from the written material for the laboratory. (Doing so constitutes plagiarism.) It is not necessary to include all of the details of the experiment. This introduction should indicate that you know the experimental quantities to be measured, the general procedures to be followed, and that you are prepared to perform the experiment.

The Laboratory Instructor will sign and date each page of the laboratory notebook each week. Give the carbon copies of your pages of data to the Laboratory Instructor at the end of the experiment. We can, and may, compare these data with the data presented in your report.

Your notebook should be neat and well organized. The procedures that you used for the notebook in QUANT Lab should be followed for the PCHEM notebook. Have data tables set up before you come to lab. Write clearly so that others can read your numbers. Do not record data on pieces of paper and then transcribe to your notebook. Mistakes (which do occur) should be crossed out neatly and the correct data written in.

Some of the experiments can be completed within one lab period, but you have two weeks to complete each experiment. The equipment for a given experiment will not be available to you in subsequent weeks because others will be doing the experiments. Most of the lab sections are full and it is not likely that you may come in at other times to complete the work. Consequently, you must complete each experiment within the allotted two weeks.

You are expected and required to attend each laboratory period, although not for the entire four-hour period. Your absence from the lab may create a problem for your lab partner. If something happens and you cannot attend your lab section, make sure that your lab partner and your Laboratory Instructor know that you will not be in lab and make arrangements to cover your work. Attendance and active participation in the laboratory experiments and the analysis of data are part of your “technique” grade.

You and your lab partner should examine your data and begin to analyze them during the second laboratory period for each experiment, if not before then. The analysis of data from these experiments is often complex and/or tedious. In analyzing your data, you may discover that you failed to record a critical parameter or that some of your data are incorrect and need to be repeated.

Housekeeping and Safety:

Clean your area before you leave the lab! After you have completed each experiment, clean the glassware and return it to the appropriate storage area. If you break anything, notify the Laboratory Instructor immediately. It takes time to get replacements. Notify the Laboratory

Instructor about any malfunctioning equipment. Six groups in different lab sections will use the equipment throughout the week.

Reasonable care should be exercised in all experiments. If you have doubts about a procedure, check with your Laboratory Instructor.

Safety goggles shall be worn in the laboratory at all times when anyone is doing an experiment.

Shorts and sandals should not be worn. You may not eat or drink anything in the lab.

Only paper – no chemicals or glass – should be put into the wastebasket. There is a separate container for broken glass and sharp plastic.

Waste containers are available for the organic compounds. The inorganic salts may be washed down the sink.

Reports:

Written reports are essential in the research and work environment. There are many types of reports whose formats are determined by the group by whom they will be read. Scientific reports are intended for other knowledgeable scientists. They should contain sufficient detail that the data can be analyzed to give the reported results and the procedures could be followed to repeat the experiment. Excessive length does not make a good report.

Your reports must be done with a word processor (WORD, WordPerfect, or any other with which you are familiar) and carefully proofread. Carelessness in writing and proofreading is often associated with carelessness in obtaining and analyzing the data. Newer programs will check spelling (a possible problem because of technical words, but you can add new words to the vocabulary) and, to a lesser extent, the grammar.

Technical reports are generally written in the passive voice. Some programs will suggest changes in this construction; ignore these suggestions. Current programs can write superscripts and subscripts, essential in chemical reports. An equation editor is also available for mathematical equations. Learn how to use these aids to write a proper report: you will do so extensively in your career.

Reports for the experiments may be written as a joint report with your lab partner and should clearly indicate that the two of you wrote the report. Both of you should be heavily involved in analyzing the data and writing the report. You will receive the same grade for the report. **However**, you may write an individual report on a jointly done experiment. Your report should clearly indicate that it is only your report, but the name of your lab partner should be included. If separate reports are written, they must not be identical. The same Laboratory Instructor will grade all of the reports for a given experiment. Comparisons of reports may be made.

Schedule for Reports:

Two weeks are allowed for the completion of each experiment. **The report on each experiment is due at the beginning of the following lab period.** The report should be given to your Laboratory Instructor at the beginning of the lab period.

If you turn in a late report, give it to one of the Laboratory Instructors, preferably during one of the six lab sections. Do not slide reports under someone's door. Do not put lab reports in the mailbox of a Laboratory Instructor. Do not give the report to someone to give to the Laboratory Instructor. Do not put the lab report on anyone's desk. If you cannot find a Laboratory Instructor, you may give your report to Ridge (106 LDL). Again, it is inadvisable to slide the report under Ridge's door or put it on his desk: the report may get lost.

A penalty of 3 points per day (excluding weekends) will be applied to late reports. No laboratories are scheduled for the last half-week of the semester, 12/7 – 12/11. The last lab report is due on the day during that week when the lab would be met. Make arrangements with your Lab Instructor to turn in your report. No lab reports will be accepted after F, 12/13.

Graded lab reports will not be returned, but will be available for your inspection, usually within one week of submission.

Form of reports:

The CHEM 445 Home Page shows a lab report from the recent past for an experiment that we no longer do.

I. Title page and abstract (10)

On a single page, give the title of the report, the names of the authors, whether this is a joint or single author report, the date due and the date of submission, and a short abstract. The abstract is a brief summary of major results of the experiment with data. The abstract is written after the data are analyzed and the report is completed.

II. Introduction (5)

Give the purpose of the experiment and a short summary of the theory. Don't copy from the written lab material (plagiarism). Don't apply "overkill". This section should be relatively short, a few hundred words, but with necessary equations.

III. Procedure (Experimental methods) (10)

Briefly describe the instrumentation and procedures that you followed. The procedures have been written in detail for you to follow, but do not include all of this material (plagiarism). If there are pertinent points that were omitted in the written procedure for the lab include them. If there are critical points in the procedure, indicate them. Again, a few hundred words should be sufficient for a general description of the procedure.

IV. Data and Results (50)

This section is the heart (or meat) of the report. Present all results from primary data to derived results – generally in tabular form. Clearly indicate the meaning of each column of data with footnotes to the tables. Give units. Tables and figures should be numbered consecutively, have descriptive headings, and appear in the body of the text, frequently as a separate page, after the first reference to the table. Do not refer to Table II in the text before referring to Table I, etc.

Do not put all of the data in a single table. There will generally be too much data for anyone to comprehend. Do not discuss any data not presented in a table. Do not present data in a table that are not discussed in the text.

Give the equations that you use for the analysis of the data. Analyze the data with a spreadsheet or other program (Excel, Mathcad, Maple, etc.) Specify the program that you used. Include sample calculations for pertinent results.

When you have multiple values for a single quantity, you should always report the average and standard deviation for the set of data. You may include the 95 % confidence interval. Harris, *Quantitative Chemical Analysis*, from CHEM 119 has appropriate equations. We will use the following definition of standard deviation, σ :

$$\sigma = \sqrt{\frac{\sum_{j=1}^N (x_j - \bar{x})^2}{N - 1}}$$

In some experiments, one estimates the uncertainty in a final measurement from the uncertainties of the individual measurements by what is often described as “propagation of error”. Harris, *QUANTITATIVE CHEMICAL ANALYSIS*, or many other books, gives standard equations for this type of analysis. Dybowski has a discussion of propagation of error on the CHEM 446 Home Page. There are Web Sites (PCHEM lab pages) that discuss propagation of error analyses in varying depths and degrees of confusion. An estimate of the uncertainty of the value you calculated is essential, but one is concerned about the precision of the number, not the precision of the uncertainty.

It is generally useful (and sometimes required) to present results in graphical form. In your reports you should also have a table of the values that you plot for each figure. Use one of the standard programs to plot your data. Label the axes, with units. Clearly identify points if more than one set of data are plotted in the same figure (a useful way of comparing data). Plots should be on separate pages in the text. Landscape printing generally allows more space for the plot but requires that the page be turned at right angles to be seen. **Portrait is fine for most plots.** Color plots are OK if they show something more obviously than black and white. It is also easy to change the shape of the points and the texture of the lines in black and white plots to indicate different sets of data. Journals don't like color plots.

When you use a computer program to estimate intercepts and slopes of equations based on experimental data, you should get estimates of the uncertainties of these values.

V. Discussion (30)

The discussion section is often combined with the results or analysis section. In these reports, it should be a separate section that includes the conclusions from the experiments and comparisons with other data. This section should have a summary of any conclusions reached earlier in the report.

For these reports, the Discussion section should include the answers to any specific questions that were asked for the experiment.

VI. References

Give the references as endnotes at the end of the report on a separate page. There are several styles that are accepted by different groups. However, when you write papers for publication, you will discover that editors (or editorial assistants) are remarkably fussy about the detailed format for references.

VII Appendices

If you have several pages of calculations or output from a computer program, put the data in the appendix. The information should be well organized and described. Don't include numbers just to pad the size of the report.

Grades:

Each lab report counts 1/7 of the grade.

The laboratory notebook and lab technique, as determined by the Laboratory Instructor, count 1/7 of the grade.

Grade	A	A-	B+	B	B-	C+
Range	≥ 93	90 – 92	87 – 89	83 – 86	79 – 82	75 – 78
Grade	C	C-	D+	D	D-	F
Range	71 – 74	67 – 70	63 – 66	59 – 62	55 – 58	≤ 54