

## CHEM 446

### Physical Chemistry Laboratory

Spring 2012

**Textbook:** There is no course text. Some experiments were originally designed around experiments in books like D. P. Shoemaker, C. W. Garland, J. W. Nibler, *Experiments in Physical Chemistry*, 6th Edition, McGraw-Hill, New York, 1996, or A. M. Halpern, *Experimental Physical Chemistry, A Laboratory Textbook*, 2<sup>nd</sup> Edition, Prentice-Hall, Englewood Cliffs, New Jersey, 1997; or J. M. White, *Physical Chemistry Laboratory Experiments*, Prentice-Hall, Englewood Cliffs, New Jersey, 1975. Our experiments are not quite identical to the experiments in those books, but you should use them as references. Where appropriate, the corresponding pages in these books are indicated as a reference for you.

#### *Sequence*

Experiments are done in rotation. Each student performs the following laboratory experiments. A schedule of laboratory experiments is posted in the laboratory and also on the Web: (<http://www.udel.edu/pchem/C446/c446.htm>). In addition, each student must do the experiment on the statistical treatment of data.

#### *Laboratory Experiments*

- Dipole Moment of Polar Molecules in Solution
- Determining the “Size” of a Molecule from Viscosity
- Conductance of Solutions (Determining the Equilibrium Constant of Acetic Acid)
- Experimental and Calculated Vibration-rotation Spectra of HCl, DCl, and CO
- Surface Tension of Pure Liquids
- Kinetics of Reduction of Methylene Blue

Information on each experiment at the University of Delaware is available as a downloadable PDF file at the course Web site.

#### *Preparation for the Laboratory*

- Prepare for each experiment carefully.
- Reading the theory and experimental procedure thoroughly before walking into the laboratory.
- Outside of class time, during the week BEFORE you are to start an experiment, you and your laboratory partner should meet to discuss the experiment. Summarize in your laboratory notebook any important points about the experiment (things like the number of values of a variable you plan to investigate, including specific numbers if possible, the possible problems you might encounter, the possible results you might expect, and possible alternate

experimental studies). The object of this exercise is to think about HOW to do the experiment BEFORE you come to laboratory. The notebook should be clear on what you intend to do and how you are going to do that.

- Six experiments are being done simultaneously. At this level of activity the instructor cannot describe each experiment at the beginning of the laboratory period. **If you are found not to be prepared, you will be asked to leave the laboratory.** Only when you have demonstrated knowledge of the experiment will you be allowed to return.

**IMPORTANT: Your laboratory notebook must be given to your instructor for grading when you turn in your last lab report.**

### *General Information*

- Much of the work requires knowledge of instruments. If you do not know how to use a particular instrument, ask! If you injure equipment, it results in a lower grade. **SIGN THE LOGBOOK** every time you use an instrument. If there is a problem, note that.

### *Record-keeping, Documentation, and Required Duties in the Laboratory*

- Recording and documenting data is essential to research. All data should be recorded in a **bound research notebook with numbered pages**, with provision for a copy of each page. The Bookstore has at least three acceptable notebook styles. Other stores may have equivalent notebooks. You may reuse your CHEM 445 notebook if it is available from last semester. We review the notebooks at the end of the semester and assign a grade, so you may not simultaneously use it in another laboratory course during this semester. **The notebook can be returned to you at the end of the semester with the pages expunged if you make a request.**
- Leave a page at the beginning of the notebook for a table of contents, which you should fill in as you go along.
- As discussed above, the laboratory notebook should contain specifics you believe important to carrying out an experiment. Since not all of the specifics of an experiment are given in the write-up, your laboratory book should reflect what you intend to do. It is also a means for the instructor to check that someone has read and understood an experiment. Describe (1) what you are trying to determine, (2) how you will determine that, and (3) any procedural problems you think might occur, including safety issues.
- The instructor **signs and dates** each page of the laboratory notebook at the end of each period. The copy of your data pages must be given to the instructor at the end of the experiment and may be used for checking the validity of information in your report.
- Most experiments may be completed within one four-hour period. You and your partner should examine and analyze your data before the second scheduled laboratory period to determine if additional experiments are needed in these cases. The equipment for an experiment is not available to you in subsequent weeks because others use it during the other weeks of the semester, so **you must finish each experiment in the allotted two weeks.**
- You may not “work ahead” as this provides scheduling problems.

- **You are expected to come to the laboratory the second week UNLESS you have turned in a laboratory report for the experiment.** Laboratory meets each week. If you have finished the work in a previous week, you must be prepared to show the instructor that you have sufficient data, that they are properly being reduced, and that you do not need to do any further experiments. It is insufficient to simply show the data sheets; you must show that you have examined the data and are actively and properly reducing data. The second laboratory period is a very good time to write the report on an experiment, as you and your partner are guaranteed to have four hours to work on it.
- If you break anything, notify the instructor immediately. It takes time to obtain replacements. Failure to notify the instructor about breakage or nonfunctioning equipment will result in a substantial deduction from your grade.
- **Clean your area when you leave!** After you have completed a day's work, clean the glassware, dry it in the oven, and return it to the appropriate storage area. Make certain it contains NO residue. Put chemicals and equipment back in the proper place. Throw away all dross. Clean the area with paper towels, if necessary. Several groups in the other laboratory section use the equipment. Failure to clean an area thoroughly results in a deduction from your grade for the experiment, if we find dirty areas when the subsequent group uses it. The presumption is that immediate preceding groups are responsible if an area (and specifically glassware) is not properly cleaned.

### **Reports**

- Written reports are essential in the research and work environments. There are many types of reports, whose formats are determined by the intended readers. Scientific reports are generally intended for other competent scientists. They must be well organized and readable. They should be **typed** and **neat** – all parts of the report!
- It is important to use proper style. Do not neglect grammar, spelling, and proper usage. Care in preparation lends credibility to a report. Conversely, errors in preparing a report tend to elicit questions about the care with which the experiment was done. There are several good sources for writing style. One source I like is by Strunk and White.<sup>1</sup> Another extremely useful book is the *ACS Style Guide*, which provides details about elements of style for publication in ACS journals.<sup>2</sup> References should be in the styles given in the *ACS Style Guide*. The important sections of the *Style Guide* can be found on the internet.
- Reports should be done with a word processor like *WORD* and carefully proofread. Newer versions of the word-processing programs check spelling (a possible problem because of technical words) and, to some extent, grammar. Use an equation editor for entry of mathematical equations, as has been done in this document.
- You should view report preparation as an exercise in learning to communicate your work, because this is very important to your career. While the proper collection and analysis of data is tremendously important, attention to presentation is also important.
- The size of the report is limited to eight pages. An acceptable format for a laboratory report is given at the Website.

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<sup>1</sup> W. Strunk, Jr. and E. B. White, *The Elements of Style*, 3<sup>rd</sup> Edition, Macmillan, New York, 1979.

<sup>2</sup> J. S. Dodd (ed.), *The ACS Style Guide*, American Chemical Society, Washington, DC, 1986.

The sections of a report are:

- **Title and Abstract**

This section contains the title of the report, the names of the authors, the section number, and the date of submission. The abstract provides a summary of the report; it is not an introduction. Do not put tables in the abstract. In general, an abstract is written **AFTER** the body of the report is completed, not before. The title and abstract should, together, be no more than one page.

- **Introduction**

Give a **short** introduction in your own words, stating the purpose of the experiment and any comments about the experiment. Do not duplicate the introductory and theoretical sections of the experimental write-up; we don't need to read things twice (particularly those we have written!) It should be no longer than approximately 200 words and should be a single paragraph.

- **Results and Discussion (including calculations and error analysis)**

This extensive section is the heart of the report. In it, the author presents all results. It should be written like the similar section of a journal article. In it, you should clearly show how and why the data were analyzed. You do not have to reproduce all data in this section. Explain what was done, how it was done, and present the results in a readable, organized format. The format of reports depend on the experiments. One should include discussion of results, explanation of problems with the experiment, or other topics of importance to the reader's understanding of the data. One common problem is proper captioning of figures and tables; make sure that everything is clearly and completely labeled. The titles of tables go above the table body, whereas the caption of a figure goes below the figure. Do not write two captions for figures. Figures should be numbered consecutively through the report, and the caption should explain any particular information needed to understand the figure.

- **Conclusion**

There should be a short concluding section that summarizes the experiment, including pertinent results.

- **References (if any)**

The references (if any) should conform to proper style for ACS publications. That style can be found in the *ACS Style Guide*. They should be collected in a section after the conclusions. Be consistent in style, whatever you do. They should be numbered consecutively through the report, although you do not need a separate number for each time the same reference is made. Do not use references to internet sites; find the original reference in a book.

- **Appendices**

Sometimes it is necessary to insert information in a report that may be useful, but which is not central to the understanding of the report. This information is to be provided in an appendix or in several appendices at the end of the report.

- **Discussion Question Answers**

In the write-ups are discussion questions about the experiment. You should provide answers to these questions in an appendix to your report entitled **Discussion Questions**. The answers should take only a sentence or two. Type the discussion question, followed by an answer.

## ***Honesty and Reporting***

Reports on experiments you carry out alone must be **your own work**. Reports on experiments done in collaboration with another student may be written as a single joint report. However, you do NOT have to prepare a report in conjunction with your partner. Work carried out jointly may be reported individually (but must include the name of your partner); sometimes it is simply not convenient for two people to get together to make a report; this is something you should discuss with your partner. Any student may choose to write individually at any time. You should, of course, inform your partner that you wish to write a separate report. If separate reports are written, they should obviously not be identical. Your data and your report (either done individually or in pairs) should not be identical to anyone else's report. The same instructor grades all reports for a given experiment. Comparisons of reports may be made. Plagiarism of any kind is unacceptable and is dealt with harshly, a grade of zero being assigned when plagiarism has been determined to have existed.

## ***Uncertainty in Quantities***

Scientific measurement is founded on reproducibility of results from laboratory to laboratory, from day to day, and from person to person. **Any reported quantity must have an associated uncertainty**. Error analysis is the subject of numerous books.<sup>3</sup> There is a discussion of uncertainty analysis in a document on the Website, which you should read carefully. **In all cases, you must give an estimate of uncertainty in any parameter whose value you quote.**

## ***Schedule for Reports***

- Two weeks are allowed for the completion of each experiment. In principle, the experimental work can be done in one laboratory period, and the second period can be used for writing the laboratory report. If necessary, you may wish to take additional data during the second period.
- The report on an experiment is due no later than the **start of the laboratory period subsequent to the second period**. You may always give us your reports early!
- **You must give laboratory reports to either your laboratory instructor or the professor in person. You may not leave them under doors or in mailboxes. Reports found on floors or in mailboxes will be discarded! It is YOUR obligation to find the proper person to receive your report.**
- Late reports lose 3 points per day (including weekends). For purposes of this requirement, the day begins at the opening moment the laboratory period in which a report is due. **Reports given after the opening of the laboratory are late.** So, do not ask to wait until you can print a report or you can finish this or that; the due time is the beginning of the laboratory, and you should finish in time to turn the report in at that moment.

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<sup>3</sup> (a) Y. Beers, *Introduction to the Theory of Error*, Addison-Wesley, Reading, MA, 1957; (b) P. R. Bevington, *Data Reduction and Error Analysis for the Physical Sciences*, McGraw-Hill, New York, 1969; (c) A. L. Edwards, *Multiple Regression and the Analysis of Variance and Covariance*, Freeman, San Francisco, 1979; (d) J. R. Green and D. Margerison, *Statistical Treatment of Experimental Data*, Elsevier, Amsterdam, 1978; (e) M. G. Natrella, *Experimental Statistics*, National Bureau of Standards Handbook 91, National Bureau of Standards, Washington, DC, 1966; (f) J. R. Taylor, *An Introduction to Error Analysis*, Second Edition, University Science Books, Sausalito, California, 1997.

- Reports more than one week late will **not** be graded, and the student will receive a grade of 0 for that report. Do not wait to turn in reports. You are given adequate time to perform experiments and write the report.
- Incomplete grades are not given in this course; all reports must be finished before the end of the semester. Any missing grades are assigned zeros and the final grade is calculated on that basis.
- No laboratories are scheduled during the last (half) week of the semester. The report on the last experiment must be given to your instructor no later than the beginning 10 minutes of the beginning time of the laboratory period on the day that the laboratory would have met.
- **No reports are accepted after the last day of classes.**
- Graded reports are not returned but are available for your inspection, usually within one week of submission. You should examine your graded reports because the comments help you write the next report in better style. Your reports are kept in a file and are available for your inspection on request.
- You may not take a report out of the laboratory for any purposes, once it is turned in.

### *Safety*

- Laboratory neatness is an essential part of laboratory safety. **Clean your area when you leave!** Other groups in the different lab sections will use the equipment.
- Reasonable care should be observed in all experiments. Consult your laboratory instructor if you have any questions.
- The cardboard waste containers are for laboratory materials; do not put laboratory materials into the trashcan.
- Waste containers are available for organic chemicals in the hood; put all waste organics in the proper container.
- Inorganic salts (but not those of heavy metals) may be washed down the sink.
- Paper, but no chemicals or glass, should be put in the wastebasket.
- **Safety goggles shall be worn in the laboratory as eye protection.**
- Shorts and sandals shall not be worn in the laboratory at any time.
- No open-toed shoes are to be worn in the laboratory at any time.
- Failure to obey common safety rules results in immediate expulsion from the laboratory room. Any missed experiments because of expulsion are graded as if they are the result of an unexcused absence and given a grade of 0.

### *Grading*

Your grade will be determined by the following general factors:

1. quality of written reports, including organization, spelling, proper grammar, and style
2. accuracy and analysis of data
3. work in the laboratory (e.g., how you carry out experiments, how you leave the laboratory space, safety, knowledge of experiments as determined in discussions, your contribution to work in a partnership, and other such criteria as the instructor may deem important)

Quantitatively, the following guide is used to assign grades at the end of the semester:

- The report on each experiment counts 1/9 of the grade.

- A laboratory-technique grade (including any quizzes, exams, and especially the notebook and record-keeping) counts 1/9 of the grade. [Do not neglect preparation and writing in the notebook, as these are part of your laboratory instructor's grade.]
- The final examination counts 1/9 of the grade.
- The following grading standard is used **for unsigned grades**.

Lowest A	90
Lowest B	80
Lowest C	70
Lowest D	60

Grades with signs (A-, B+, etc.) are assigned in the intermediate ranges, at the instructor's discretion, but the above lowest grades are guaranteed.