Chemistry 125: Advanced Methods in Physical Chemistry

Texts: Shoemaker, D. P.; Garland, C. W.; Nibler, J. W. *Experiments in Physical Chemistry*, 7th ed.; McGraw–Hill: 2003. ISBN: 0–07–231821–X. Any edition will work for this class.

Skoog, D. A.; Holler, F. J.; Nieman, T. A. *Principles of Instrumental Analysis*, 5th ed.; Saunders: 1998. ISBN: 0–03–002078–6. Any edition will work for this class.

Lecture Topics and Assignments

The lectures will deal with the chemical literature, error analysis, and computer interfacing. Readings and problem sets covering these areas will be assigned periodically.

Laboratory Topics and Assignments

Everyone is required to do both the Fourier transform infrared based measurement of the vibrational–rotational spectra for HCl and DCl and the Matlab lab. Your group assigned by the instructor will choose its preference for the third lab, which you will have the remainder of the quarter to complete.

Before beginning each experiment, you must have in your notebook a brief written experimental plan that your TA must approve. This plan should indicate what you intend to measure, how you are going to perform the measurement, what equipment you will need, what safety precautions you will take, what data must be taken, etc. The TA will determine whether or not the plan is adequate for you to begin the experiment. You will not be permitted to begin any experiment without this TA approval.

You are required to collect thorough data sets and write extensive reports on all of your experiments. A data set will typically involve duplicate or triplicate measurements and multiple variations in experimental parameters, such as concentration, temperature, instrumental settings, etc. Due dates for the lab reports are TBD and will be announced in class and on Canvas. The expected format for your reports is described below.

In addition to your lab reports, each laboratory group will be required to present the results of the third lab as an oral report to the class during finals week. The talk should be approximately 25 minutes in duration, followed by 5 minutes for questions. The use of PowerPoint to prepare your presentations is highly recommended. The format of your talks should be similar to that of an ACS National Meeting.

All three experiments and the oral presentation must be completed, and all of the written reports submitted, in order to pass the course. Any late reports will be subject to a penalty proportional to the number of days tardy. The penalty is 5% of the possible score for each day late. No work will be accepted after the last day of lab.

All of your experiments will be done with lab partners. You are encouraged to discuss your data and its analysis and interpretation with your lab partners, other students, and the TAs. However, the actual data analyses and the written reports must be done entirely independently of your lab partners or other students (past or present). Make sure that you avoid unauthorized collaboration and plagiarism. All suspected violations of the Code of Academic Conduct will be referred to Student Judicial Affairs.

Grading

Your written laboratory reports make the largest contribution to your grade. These will be scored in terms of presentation; thoroughness of the experimental data; quality of the data analysis and interpretation; and the insight demonstrated and conclusions reached. A rough break down of the various components in your final grade follows.

Reports (3×100)	300	(60 %)
Oral presentation (1 x 100)	100	(20 %)
Standard Operating Procedure	50	(10 %)
Instructor and TA evaluation	50	(10 %)

Laboratory Reports

The reports must be typed including formulas (MathType is recommended). The textual material (not counting tables and figures) should be 6 – 10 double-spaced pages in length. The reports must be written in the style of an ACS journal article. You should examine recent journal papers from *J. Phys. Chem.* or *J. Am. Chem. Soc.* (see electronic journals, staring at http://www.lib.ucdavis.edu/pse/), or *The ACS Style Guide* (see reference stacks at PSE Library, call number QD8.5. A25 1997), for the accepted style. In general, the following sections, each clearly labeled and in the order given, are required.

Abstract. This consists of a concise summary of your results in less than 100 words and in the present tense.

Introduction. Give the main purpose of the experiment, any relevant background, the methodology used, and a brief general statement of the results obtained and the conclusions.

Theory. Present the required theory to understand the experiment, assuming that the reader's knowledge is commensurate with Chemistry 110. It should be complete in terms of the needed equations to interpret your results, but not overly verbose. Derivations should be referenced, not given in detail.

Experimental. Indicate exactly how the experiment was done. A reference to a text or journal article may be sufficient. However, state any modifications you made to a referenced procedure. Be explicit as to types and sources of chemicals, amounts used, instruments employed, etc.

Results. Present your results in prose form, as in a journal article, not just as a collection of tables. Number any accompanying tables or figures (graphs) consecutively and refer to them by number in your writing. All numbers (in the text, tables or graphs) should have the correct number of significant figures, associated error limits (estimated standard deviations or confidence limits) and SI units. Also each table should have a title and each figure a figure caption much like those in journal articles.

Discussion. Discuss your results and present the conclusions. Compare your results to literature values. If these are unavailable for your exact results, then compare with similar results in similar systems. The latter are always available. In so far as reasonable, you should also calculate theoretical estimates for comparison with your experimental results, particularly if commensurate with Chemistry 110. Address the significance of your results in terms of the chemical system studied.

Error Analysis. Present a detailed sample calculation, showing in particular how you estimated the random errors in your measured quantities, and how these errors were propagated to obtain the errors quoted in your final result. Also, discuss possible systematic errors and how these would effect your results. Give suggestions for reducing the errors or otherwise improving the experiment.

References. You should have multiple references to citations other than your textbooks. The accepted format is given in *The ACS Style Guide*, and in "Notice to Authors" from *J. Phys. Chem.* (see electronic journals, starting at http://www.lib.ucdavis.edu/pse/).

Laboratory Rules

The following rules are mostly for your own good as well as common courtesy to others in laboratory.

Safety. Be careful and think before you act. Many of the chemicals and items of equipment that you will be using are potentially hazardous. If unsure, ask a TA before proceeding. You are required to wear suitable clothing and safety goggles at all times while in lab. Eating or drinking is prohibited in lab.

Waste. Hoods in the Annex laboratories have containers that are labeled for specific types of chemical waste. You must use the appropriate one of these containers to dispose of any chemical waste that cannot be safely poured down the sink. If unsure, ask a TA before proceeding. Specific instructions for the disposal of particular chemicals are included in some of the experimental instructions or handouts.

Storeroom. Check with the TA before going to the second floor storeroom to request any equipment or chemicals. Nearly everything that you will need is available in the laboratory. Also, inform the TA of anything that is broken or otherwise inoperative. We don't care how it got that way (up to a point), but we want to have it repaired before someone else needs it.

Instruments. Be sure you know how to operate an instrument before attempting to do so. Read any available manuals, and consult the TA if necessary. Never be afraid to ask questions, particularly when high voltage or current, fragile glassware or expensive instruments are involved.

Clean Up and Security. It is your responsibility to be sure that your set-up is secure and safe when you leave each day. Clean up and disassemble each experiment when you are done unless explicitly instructed not to do so by the TA.

Laboratory Experiments

Following is an incomplete list of available laboratory equipment for this quarter and some literature references. Copies of references which are not from Shoemaker *et al*. are available on the course web page. Everyone must complete the Fourier transform infrared measurement of the vibrational–rotational spectra of HCl and DCl as well as the Matlab lab.

1. *Fourier transform infrared spectroscopy*

The high resolution vibrational spectra of gaseous HCl and DCl are obtained using a Fourier transform IR

spectrometer. An assignment of the spectra and analysis of the frequencies will yield the rotational constants, bond lengths, force constants and other parameters. The isotope effect on these is also examined. The results are compared with the predictions of electronic structure calculations.

Ref: Shoemaker *et al*., Experiment 37
Schwenz, R.W.; Polik, W.F. *J. Chem. Educ*. **1999**, 7*6*, 1302.
Prais, M.G. *J. Chem. Educ*. **1986**, *63*, 747.
Perkins, W.D. *J. Chem. Educ*. **1986**, *63*, A5.

2. *Matlab lab*

An understanding of basic error analysis, the reduction of experimental data to molecular parameters, and the skills associated with writing are central to physical chemistry. This laboratory uses a Matlab macro to generate artificial spectral data that will be analyzed using Matlab data reduction routines developed in class.

3. *Visible absorption spectroscopy*

Ref: Shoemaker *et al*., Experiment 40
Snadden, R.B. *J. Chem. Educ*. **1987**, *64*, 919.
McNaught, I.J. *J. Chem. Educ*. **1980**, *57*, 101.
Stafford, F.E. *J. Chem. Educ*. **1962**, *39*, 626.

4. *Ultraviolet/visible spectroscopy*

Ref: Daniels *et al*., *Experimental Physical Chemistry*, Experiment 25.
Birk, J.P.; Walters, D.C. *J. Chem. Educ*. **1992**, *69*, 585.
Chem. 125 SmartSite

5. *Nuclear magnetic resonance*

Ref: Morris, K.F.; Erickson, L.E. *J. Chem. Educ*. **1996**, *73*, 471.
Gasparro, F.P.; Kolodny, N.H. *J. Chem. Educ*. **1977**, *54*, 258.

6. *GC/MS/MS*

7. *LC/MS/MS*

8. *Ion Mobility Spectroscopy*