

CHM4411L, Physical Chemistry Laboratory Spring 2014

Instructor: Dr. Benjamin J. Killian, LEI 202A, 392-0528, killian@chem.ufl.edu
Office Hours: M 4th Per.; W, 6th Per.; R, 3rd Per.; F 4th Per.

Teaching Assistants: Joe Duchene, CLB 406, jduchene@chem.ufl.edu
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Objectives: CHM 4411L students learn:

- Physical measurements of thermodynamic, kinetic, and spectroscopic properties.
- Error analysis and critical examination of experimental data.
- Preparation of formal written reports and oral presentations.

Texts: Killian, B. J. *Experiments for Physical Chemistry Laboratory*, Spring 2014, Target Copy; Gainesville, 2014
Williams, K. R. *Error Analysis in Physical and Analytical Chemistry*, 3rd Ed, Target Copy; Gainesville, 2008

Other Required Materials: Laboratory Notebook with duplicate pre-numbered pages; safety glasses; departmentally approved attire (long, loose-fitting pants, full shirt, shoes which cover the feet, tieback for long hair); USB drive.

Grade Distribution:

Problem Set:	1 Error Analysis Problem Set @ 100 pts (Due 1/21)	100 pts
Written Quiz:	1 pK_a Quiz	50 pts
Written Reports:	4 Full Reports (NMR, IR, LIF, pK_a) @ 100 pts	400 pts
	5 Abbreviated Reports/Handouts @ 50 pts	250 pts
Oral Report:	1 @ 75 pts	75 pts
Pre-Lab Quizzes:	9 @ 20 pts (none for Surface Analysis)	180 pts
Data Grade:	9 @ 10 pts	90 pts
Subjective Grade:		100 pts
Total		1245 pts

Factors affecting the subjective grade will be the student's attendance record (lecture and lab), preparation for laboratory work, laboratory technique, understanding of the experiments, and general attitude. Ten (10) subjective points will be deducted for each unexcused absence from lecture. Notebook spot checks will be made randomly to check for written procedures and data collection.

Students are expected to arrive on time for all lectures and labs. Attendance will be recorded at the start of lecture. Pre-lab exercises must be submitted at the start of the laboratory period. No pre-lab assignments will be accepted after the beginning of lab.

Grading Scale:

Letter Grade	Percentage	Letter Grade	Percentage	Letter Grade	Percentage
A	> 90	B-	> 77	D+	> 57
A-	> 87	C+	> 72	D	> 54
B+	> 84	C	> 67	E	< 54
B	> 80	C-	> 64		

Note: Chemistry majors earning grades below C (C-, D+, D, or E) must repeat the course to earn credit towards the degree.

Pre-Lab Assignments: For all experiments (excluding pK_a and Molecular Modeling) students will complete a pre-lab assignment. The pre-lab assignment can be found in the lab manual at the end of the experimental write-up. Students are expected to work independently on pre-labs. Pre-labs do not need to be typed; however, please follow the same presentation guidelines as with reports; i.e., include units, format LLS equations, proper graph construction, etc. When a spreadsheet assignment is completed, please turn in a formatted print-out of your results and bring an electronic version of the spreadsheet to lab with you. *You need to provide sample calculations, even when the pre-lab involves a spreadsheet layout.* Pre-lab assignments are due at 12:50 PM on the day of the experiment. NO late pre-lab assignments will be accepted.

Written Reports: It is expected that reports will be neat and written in good English, with proper attention paid to paragraph structure, grammar, spelling, etc. Substandard reports will be rewritten (with appropriate point deductions). Except for equations, laboratory reports must be typed (double space, except for the abstract and captions) using a minimum font size of 10 point. Please use a professional font, such as Times New Roman, Arial, Calibri, etc. Equations (both mathematical and chemical) should be generated using an equation editor. You may write equations by hand using black or blue ink; however, they must be clearly written with sufficient space to set them off from the text. Reports should contain the sections described below.

Full Reports

- I. Abstract: The abstract (200 words max) should give a summary of the entire experiment: what was measured, experimental method, results with 95% confidence limits. The abstract must be complete in itself, although it is separate from the rest of the paper. Place the abstract on the title page, separate from the remainder of the report.
- II. Introduction: This section should answer the questions: 1) What is the reason for performing this experiment, 2) What is the theory supporting the experiment? and 3) What is the methodology used? The second point will constitute the major part of the introduction. Give equations unique to the experiment and conditions necessary for the equations to be valid. Identify all variables with their units. Number equations consecutively in the right margin, and refer to the original number if the equation is used again (e.g., in the Sample Calculations). Also include molecular structures for pertinent compounds (other than macromolecules). In writing this section, you should aim to explain the experiment to a senior-level chemistry student who has taken physical chemistry, but who is otherwise unfamiliar with the procedure or method. Do not regurgitate the introductory material in the text or handout.
- III. Experimental Procedure: The procedure should be clearly explained *in paragraph form* with sufficient detail that a person trained on the instrument could repeat the experiment, but do not give specific commands that were used. Use the passive voice (no commands), write in the past tense (tell me what you did), and do not write in the first person. Include the manufacturer and model name/number for each instrument used and important experimental parameters. Also, be sure to include a reference to the laboratory manual.
- IV. Sample Calculations: Give sample calculations with actual data (including units) to show how results are obtained. Include text (in paragraph form) to explain what is involved; do not just write out a series of equations. Do not use headers for your calculations in a full report.
- V. Error Analysis: Include a mathematical analysis (including units) of the random errors in the experiment. Include text (in paragraph form) to explain what is involved; do not just write out a series of equations. Do not use headers for your error analysis in a full report.
- VI. Data and Results: Use a spreadsheet program (e.g. Excel) to prepare graphs, making sure that you obey general rules for graph drawing. Tables should contain all necessary data and results, but not raw (uncorrected) data or intermediate calculations (include the data needed to obtain the final presented results only; i.e., no spreadsheet dumps). Guidelines for tables and graphs are given in Young, V. *Laboratory Manual for Introductory Analytical Chemistry*, pp. 5-8 (or older versions). Also include introductory text to direct the reader to the tables and graphs, to specify experimental parameters

(e.g., temperature) held constant throughout the experiment, and to explain symbols, etc. The table summarizing the final results should include (and cite) available literature and/or theoretical values.

- VII. **Conclusions and Discussion:** This is a very important section of the report and should not be taken lightly. Referring to your summary table, evaluate the quality of your results (i.e., Do the error limits for the measured value include the accepted value?). Relate the experimental results to the chemistry of the system (What did you learn from the experiment?). Discuss pertinent sources of error (both random and systematic) and their effects on the results. If possible, make suggestions for improvements.
- VIII. **References:** Special procedures, literature values, and discussions of previous research results must be referenced in the text using superscript numbers. The references themselves belong in a separate section at the end of the report using the format specified in the ACS Style Guide, which can be accessed at <http://jchemed.chem.wisc.edu/Journal/Authors/References.html>. Note: The laboratory manual should be referenced as shown on the first page of this syllabus.

Abbreviated Reports

- I. **Abstract:** Same as full reports.
- II. **Experimental Procedure:** Reference the procedure in the laboratory manual and state any alterations. For each instrument, give the manufacturer's name and model number, as well as important settings and parameters.
- III. **Sample Calculations:** Give sample calculations using actual data and units. Give an appropriate header for each, but no additional text is required.
- IV. **Error Analysis:** Show necessary mathematical operations using actual data and units. Give an appropriate header for each, but no additional text is required.
- V. **Data and Results:** Same as full reports.
- VI. **Conclusion and Discussion:** Same as full reports.
- VII. **References:** Same as full reports.

Library Course Guide: Marston Science Library has a number of important references available to you, including a course guide that can be found at <http://guides.uflib.ufl.edu/chm4411L>. The chemistry librarian, Dr. Neelam Bharti, is available for research questions.

Chemical Structures and Equations: It is strongly recommended that you use an electronic means of generating chemical structures and equations. Microsoft Office has a built-in equation editor. Several computer programs exist for building chemical structures. Symyx offers free academic software for generating chemical structures at <http://www.symyx.com/downloads/downloadable/index.jsp>. Note: you must register to download. Also, it is advised that you download a free chemistry font for use in your word processor. One example is the Royal Society of Chemistry font, a True-Type font that includes various symbols. It can be found at <http://www.rsc.org/education/teachers/learnnet/RSCfont.htm>.

Oral Reports: Toward the end of the semester, you will present a 20 minute talk on one of the experiments you performed during the semester. This talk is designed to mimic a professional seminar. More details will follow.

Additional Grading Policies: Written reports must be submitted by 12:50 PM on the designated dates. All assignments submitted after 12:50 PM are considered late. Deductions at the rate of 5% per day (including weekends) will be assessed for late work. The maximum allowable late time is one calendar week, after which a grade of zero will be assigned. All written work (late or otherwise) must be received by 12:50 PM on Wednesday, 04/23/2014.

Any reports that are deemed substandard or ungradable will be returned to student for revision and resubmission. The standard late deductions will apply.

Students with Disabilities: Appropriate accommodations will be provided, according to the policy at www.chem.ufl.edu/~itl/disabilities.html.

Academic Honesty: Students are expected to obey the University of Florida Honor Code, detailed at www.chem.ufl.edu/~itl/honor.html. Violations will be reported to the Office of Student Judicial Affairs.

The sale or transfer of graded or ungraded course materials to another student for use in this course (current or future semesters) is in violation of the Honor Code. All violations will be reported.

Schedule of Experiments and Reports:

Lecture		Laboratory			
Date	Topic	Date	Experiment	Report Type	Date Due
1/10	Error / NMR	1/6,7	Error Analysis	N/A	1/13,14
1/17	Bomb / DTNB	1/13,14	Keto-Enol	FULL	1/24
1/24	DTNB / Conductance	1/20,21	NO LAB (MLK Day)	N/A	N/A
1/31	Conductance / IR	1/27,28	Bomb Calorimetry (A) DTNB Kinetics (A) Conductance (B)	ABBR	2/3,4
2/7	IR / pK_a	2/3,4	Bomb Calorimetry (A) DTNB Kinetics (A) Conductance (B)	ABBR	2/10,11
2/14	pK_a	2/10,11	Bomb Calorimetry (A) DTNB Kinetics (A) Conductance (B)	ABBR	2/17,18
2/21	pK_a Quiz	2/17,18	IR of HCl/DCI	FULL	2/24,25
2/28	Phase Diagram	2/24,25	pK_a of Neutral Red	FULL	3/10,11
3/7	NO LECTURE	3/3,4	NO LAB (Spring Break)	N/A	N/A
3/14	Phase Diag. / LIF	3/10,11	Molecular Modeling	HAND-OUT	3/10,11
3/21	LIF / Raman	3/17,18	Laser-Induced Fluorescence Phase Diagram (B)	FULL ABBR	3/24,25
3/28	Raman / Nuclear	3/24,25	Laser-Induced Fluorescence Phase Diagram (B)	FULL ABBR	3/31,4/1
4/4	Nuclear	3/31,4/1	Raman Spectroscopy	ABBR [†]	4/7,8
4/11	Dosimetry	4/7,8	Fricke Dosimetry	ABBR	4/14,15
4/18	NO LECTURE	4/14,15	NO LAB	N/A	N/A
4/25	NO LECTURE	4/21,22	ORAL REPORTS	N/A	N/A

[†] The Raman Spectroscopy report will be written in the notebook and submitted before leaving lab.