# CHM 4411L, Physical Chemistry Laboratory
# Spring 2012

**Instructor:** Dr. Benjamin J. Killian, LEI 202A, 392-0528, killian@chem.ufl.edu
Office Hours: Mon, 4th Pd; Wed 4th Pd; Thurs, 7th Pd; Fri, 3rd Pd

**Teaching Assistants:** Johan Galindo-Cruz, cruz@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:** Williams, K.R. *Error Analysis in Physical and Analytical Chemistry*, Target Copy: Gainesville, 2008.

Killian, B. J. *Experiments for Physical Chemistry Laboratory*, Spring 2012, Target Copy: Gainesville, 2012.

**Other Required Materials:** Laboratory Notebook with duplicate pre-numbered pages; safety glasses; departmentally approved attire; diskette or memory stick

**Grade Distribution**:

| | | |
|---|---|---|
| Problem Set: | 1 Error Analysis Problem Set @ 50 pts. | 50 pts. |
| Written Reports: | 3 Full reports (NMR, Conductivity, and either LIF or HCl/DCl) @ 100 pts. | 300 pts. |
| | 8 Abbreviated reports @ 50 pts | 400 pts. |
| Oral Report: | 1 @ 50 pts | 50 pts. |
| Pre-lab Exercise: | 11 @ 25 pts (none for Molecular Modeling) | 275 pts. |
| Subjective Grade: | | 100 pts. |
| **Total** | | **1175 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** (in % using usual rounding conventions for fractions):

| 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 and Discussions:** For all experiments (excluding 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. 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 or Arial. 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, 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. 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 calculations 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* (see http://jchemed.chem.wisc.edu/Journal/Authors/References.html for details). 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. Calculations: Give sample calculations using actual data and units. Give an appropriate header for each.

IV. Error Analysis: Show necessary mathematical operations using actual data and units. Give an appropriate header for each.

V. Data and Results: Same as full reports.

VI. Conclusion and Discussion: Same as full reports.

VII. References: Same as full reports.

**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, found at http://www.rsc.org/education/teachers/learnnet/RSCfont.htm.

**Oral Reports**: There will an oral presentation at the end of the semester. Organize your talk to fit a 20-minute time block (typical length at an ACS meeting, etc.) and use transparencies or PowerPoint to facilitate the presentation. You should approach an oral report as a job or graduate school interview and dress accordingly. More details will follow.

**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:** Written reports must be submitted by 12:50 PM on the designated dates. 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. All written work (late or otherwise) must be received by noon Friday, 4/13/2012.

| Lecture | | Laboratory | | | |
|---|---|---|---|---|---|
| Date | Topic | Date | Experiment | Report Type | Date Due |
| 1/13 | Library Lecture (MSL 308) | 1/9,10 | Cp/Cv and Error Analysis Lecture | Abbr | 1/17 (both sections) |
| 1/20 | Error Analysis/Calor. | 1/16,17 | NO LAB | | |
| 1/27 | Calor./Kinetics | 1/23,24 | Keto-Enol | Full | 1/30,31 |
| 2/3 | Kinetics/Conductivity | 1/30,31 | Calorimetry or Vacuum Techniques | Abbr | 2/6,7 |
| 2/10 | Conductivity/LIF | 2/6,7 | Calorimetry or Vacuum Techniques | Abbr | 2/13,14 |
| 2/17 | LIF | 2/13,14 | Kinetics or Conductivity | Both[a] | 2/20,21 |
| 2/24 | LIF/IR | 2/20,21 | Kinetics or Conductivity | Both[a] | 2/27,28 |
| 3/2 | IR/Phase Diagram | 2/27,28 | LIF and IR of HCl/DCl | Full[b] | 3/12,13 |
| 3/9 | NO LECTURE | 3/5,6 | NO LAB | | |
| 3/16 | Phase Diagram/ Raman | 3/12,13 | Molecular Modeling | Handout | 3/12,13 |
| 3/23 | Raman | 3/19,20 | Phase Diagram or Raman | Abbr[c] | 3/26,27 |
| 3/30 | Dosimetry | 3/26.27 | Phase Diagram or Raman | Abbr[c] | 4/2,3 |
| 4/6 | NO LECTURE | 4/2,3 | Fricke Dosimetry | Abbr | 4/11 (both sections) |
| 4/13 | Orals | 4/9,10 | NO LAB | | |
| 4/20 | NO LECTURE | 4/16,17 | Oral Reports | | |
| 4/27 | Reading Day | 4/23,24 | NO LAB | | |

[a]Kinetics will be abbreviated, conductivity will be full.

[b]Half of the students will write up LIF; the other half will write up HCl/DCl.

[c]Raman report will be completed in lab.