

CHEMISTRY 464 – FALL 2013 – EXPERIMENTAL CHEMISTRY II

Course Information - Experimental Chemistry II, CH 464 & 464H (WIC) is the senior-level integrated laboratory course and one of the three WIC courses offered for chemistry majors and certain other science majors who meet the course requirements. Typically three main projects are performed in two lab sessions with one lecture each week. A passing grade for 3 credits requires successful completion of all experiments and all reports, as the main evaluation of student performance. All reports are individual efforts but during the lab meeting you will work in groups.

Instructor – Dr. Chong Fang Chong.Fang@oregonstate.edu
LPSC 255

IL Program Director – Dr. Christine Pastorek Christine.Pastorek@oregonstate.edu
Gilbert 247

Graduate Teaching Assistants –

Francis Han [hanf@onid.orst.edu]; Lei Chen [chenlei@onid.orst.edu];
and Liang Wang [wanglia@onid.orst.edu]

SYLLABUS Fall 2013

Course Projects -

Project 1 – Synthesis and Spectroscopic Characterization of CdSe Quantum Dots (approximately 2 weeks)

- Synthesis of CdSe nanoparticles of various sizes.
- Absorption/emission spectroscopy measurements.
- Comparison of theoretical models of the spectra of quantum dots with experiment.

Project 2 – Synthesis and Equilibrium Dynamics of HCl, DCl, DBr, and HBr (approximately 4 weeks)

- Preparation of DCl, DBr using vacuum-line techniques.
- Refraction index and dipole moment determination for HCl.
- High resolution vibrational-rotational FTIR spectra of HCl, DCl, DBr, and HBr.
- Raman determination of K_p for the H/D exchange reaction of HBr with DCl.
- Comparison of measured values of HCl properties with those from computational chemistry determined by *ab initio* quantum methods (Gaussian calculations).

Project 3 – Microlithography (approximately 2 weeks)

- Fabrication of lithography masters.
- Preparation of PDMS stamps.
- Soft lithography by replica molding and micromolding in capillaries.

d) Microcontact printing and etching.

Reports –

You are required to keep a research-style laboratory notebook with duplicate pages and to record all your experimental conditions and original observations while working in the lab. This is required writing each week but it is minimally graded. The point is to form a good habit for your future scientific research work. The directions for keeping a laboratory notebook are given at the end of this document and you are encouraged to peruse the research notebooks from Linus Pauling (one of our most famous alumni from OSU Chemistry) online at: <http://osulibrary.orst.edu/specialcollections/rnb/index.html>.

The written work required for this writing intensive course fall into the following types:

A. Full Written Reports include the following sections (the % numbers in parentheses indicate the portion of the mark of the report assigned to each section):

I. (5%) **Abstract:** Summarize the main findings and perspective of the experiment.

II. (10%) **Introduction:** Explain the objectives and rationale of the experiment.

III. (10%) **Experimental:** Briefly describe the instrument, summarize important reactions involved and changes from published procedure (sufficiently that a layman could understand what was done).

IV. (15%) **Results:** Present your results in tabular and/or graphic form as appropriate. You may use printouts of sections of your spread sheet calculations but make sure that headings are provided so that it is clear what the numbers represent.

V. (20%) **Calculations:** Use complete sentences to explain calculations. Give equations and sample calculations. If in doubt about a result, see the instructor before submitting your report. (Don't wait until the last minute!)

VI. (10%) **Errors:** Give the standard errors from any least squares fits of your experimental data. Beyond this, briefly discuss where you can the likely errors involved in the measurements and their effect on your results.

VII. (25%) **Discussion:** This is the most important section of the report since it gives you an opportunity to show originality in thinking about the results and in offering your interpretations. Explain clearly the significance of the results and offer comparisons with primary literature values for data and results on similar molecules if possible. *Answer all questions posed in the manual.* Discussion of problems encountered and possible improvements or extensions in the experiment is encouraged.

VIII. (5%) **Primary and Secondary References:** List primary and secondary references from the scientific literature (not the lab manual) used in the preparation of

your report using journal style format (see for example, Chapter 6 of the ACS Style Guide).

Full reports should be complete but concise and conform to the normal standards of scientific journal papers. They should be printed with all necessary graphs, sensible portions of spreadsheet printouts etc. fully labeled and explained. You will have the opportunity to revise some of your writing after the instructor has reviewed and commented on it during the term. This provides some meaningful research experience.

B. Short Reports that encompass some fraction of a full report.

C. Spreadsheet Exercises.

D. Data Summary Sheets.

Grading –

A passing grade requires successful completion of *all experiments and all reports*. All reports are individual efforts but during the lab meeting you will work in groups and active discussions are encouraged between partners.

Grading emphasizes accurate measurements and detailed answers/reasoning to questions posed in the lab manual. The overall grade for the course is based on the written laboratory reports as shown below, which is also directly related to your lab performance.

Late reports will receive a 10% decrease in maximum points allotted per calendar day late.

Project 1 - Synthesis and Spectroscopic Characterization of CdSe Quantum Dots

Spread Sheet Exercise #1 (5 pts)

Full Report (35 pts)

TOTAL: 40 pts

Project 2 – Synthesis and Equilibrium Dynamics of HCl, DCl, DBr, and HBr

Spread Sheet Exercises #2 & #3 (10 pts)

Synthesis Summary Sheet (5 pts)

Short Report: Refraction Index of HCl (20 pts)

Short Report: Dipole Moment of HCl (20 pts)

Full Report: IR Vibration-Rotation Analysis (45 pts)

Raman/Equilibrium Constant Summary Sheet (10 pts)

Computational Chemistry Summary Sheet (10 pts)

TOTAL: 120 pts

Project 3 - Microlithography

Short Report (30 pts)

TOTAL: 30 pts

WIC Statement

This is a Writing Intensive Course and satisfies the WIC requirement for majors in the Department of Chemistry. Students from other majors and graduate students who elect to take the course are expected to meet all course requirements, including writing. **However, this course does not satisfy the WIC requirement for majors other than Chemistry.** Because this is a writing intensive course, students will:

- use writing as a way of learning course material
- learn and practice writing as professionals in the field of scientific research/composition/education
- complete informal, ungraded or minimally graded writing assignments
- complete formal, graded writing assignments which are taken through the full writing process, including drafts and revisions
- receive and give peer feedback on writing in progress
- revise and polish at least 2000 words of writing (8-10 pages)
- complete a total of at least 5000 words of assigned writing.

Texts and Learning Resources:

The Chemistry Department Writing Guide^{III} should be consulted as an overall guide to professional writing in chemistry. It is free and posted on the web (turn to the Writing Guide link from Courses & Seminars on the main chemistry department home page: <http://www.chemistry.oregonstate.edu/>).

The ACS Style Guide, A manual for Authors and Editors, Janet S. Dodd, Ed., American Chemical Society, 2nd Ed, 1997, is a required text for this course. This is a reference on professional writing for chemists.

Any physical chemistry text such as Atkins or Alberty (whatever text you used in CH 440).

Experiments in Physical Chemistry, Garland, Nibler and Shoemaker, 7th Ed., McGraw Hill, 2003.

Laboratory Manual for Experimental Chemistry II, CH 464 & 464H (WIC) – handouts on experimental procedure and questions to be answered in the report.

Additional Reference Materials: There are additional reference materials for this class in a black notebook in room GBAD 311 (2 copies). Please do not remove except for short term copying purposes! Thank you!

General reading on molecular spectroscopy:

1. G. M. Barrow, "Introduction to Molecular Spectroscopy", McGraw-Hill (1962). (Also, any Phys. Chem. Text will give some background material).
2. J. L. Hollenberg, "Energy States of Molecules", J. Chem. Ed. 47, 2 (1970).

Vibration-rotation constants for diatomic molecules such as HCl, DCl, HBr, DBr, I₂.

3. Tables from
 - a) Huber and Herzberg, "Constants of Diatomic Molecules", D. Van Nostrand (1979).
 - b) Herzberg, "Spectra of Diatomic Molecules", D. Van Nostrand (1950).

Dipole moment data:

4. N.B.S. circular NSRDS-NBS 10 available in library. Copies of pages relevant for HCl are in the reprint booklet. Now also available online at <http://www.nist.gov/data/nsrds/NSRDS-NBS-10.pdf>.
5. D. A. Coe and J. W. Nibler, "Gas Phase Dipole Moment and Refractive Index Determinations: Two Physical Chemistry Laboratory Experiments", J. Chem. Ed., 50, 82 (1973).

References on Linear Regression:

6. Theory and Equations - GNS and also "Data Reduction and Error Analysis for the Physical Sciences" by P. R. Bevington (McGraw Hill)
7. Linear Regression procedures in Excel - Michael L. Orlov (OSU Ph.D. 1998) "Multiple Linear Regression Analysis Using Microsoft Excel"

Student Learning Outcomes

1. Develop and practice observational skills:

- Develop observational skills for making discoveries in the laboratory
- Recognize when an experiment is not working and be able to adjust amounts of reagents, conditions, equipment, etc., to solve the problems and make the experiment work successfully

- Record observations in a logical order in research-styled notebook while performing the experiment in the laboratory
- Document experimental and theoretical work including laboratory procedures, experimental conditions, materials used, equipment or software used, data and the results
- Respect and acknowledge the intellectual property of others.

2. Prepare for laboratory work:

- Come to lab prepared to work and plan to make the most of your laboratory experience
- Read and study the laboratory manual and other reading assignments carefully
- Research topics that are unknown to you by studying the published scientific literature at the library and online
- Research and document laboratory cautions and hazards before starting lab work by consulting standard references such as the CRC, MSDS, and Merck Index
- Plan experiments wisely so that you can work carefully, efficiently and successfully.

3. Prepare professional scientific reports:

- Recognize that writing is a serious process that develops from the practice of writing draft and revision copies to produce a final polished report, intelligent and understandable
- Compose and generate professional scientific reports that include well-crafted sections on: abstract, introduction, experimental methods, results, discussion, conclusion, references, supplemental information if applicable
- Develop a concise scientific writing style that is suitable for publication by practice and example from the literature, and with guidance from peer and instructor review
- Critically review others' work and offer constructive suggestions for improvement on style and content
- Communicate with instructor for their feedback on how to improve report writing, particular on problem solving and critical thinking
- Perform a statistical analysis of your results and calculate confidence limits to describe the precision and accuracy of your data
- Compare and contrast your results with published results and use sound scientific principles as a basis for justifying differences, and offering suggestions for future improvement if applicable
- Prepare reports using a word-based software

- Prepare proper scientific tables, figures and charts using a spreadsheet and learn how to incorporate these data directly into the report; create visually appealing and scientifically robust illustrations with softwares
- Use commercial drawing programs like ISIS draw or ChemWindows to draw chemical structures and reaction schemes and import into reports
- Critically evaluate the quality of the experimental results and suggest what improvements might be made in the future.

4. Operate scientific instruments & softwares:

- Operate a variety of scientific instruments and equipment from start to finish to get meaningful scientific data. Examples of the scientific instruments available for use in Experimental Chemistry II CH 464 & 464H are: FTIR, dipole moment system, high vacuum lines, diode array UV/Visible, Fluorometer, thermocouples, He-Ne laser, atomic force microscope (AFM), and SPEX scanning monochromator, etc.
- Recognize typical output from each instrument and be able to optimize adjustments in gain, detector voltage, scan range, etc., to produce optimized output
- Use specialized instrument software to operate spectrometers and set instrument controls, such as slits, band pass, sensitivity, and select the output mode
- Learn to use computational chemistry software programs, such as Gaussian 03 and HyperChem, to predict geometries and energies for molecules using semi-empirical and *ab initio* theory
- Recognize the performance characteristics for the types of cells and solvents used in each instrument
- Recognize the useful concentration ranges for each analysis and know the limitations of each instrument
- Operate a mechanical vacuum pump to produce a high vacuum
- Use cryogenics and temperature baths to adjust the temperature of an instrument or cell
- Use compressed gas cylinders equipped with regulators safely
- Adjust stopcocks on a high vacuum line to control the vacuum and flow of gases.

5. Develop teamwork to divide project duties ensuring efficiency and quality of the final results:

- Be an active contributor to your group with intelligence and good heart
- Work together for the common good of the project and meet designated schedules
- Respect the opinions of others and recognize that different perspectives on a situation can lead to an improved understanding of the problem at hand.

6. Design templates using spreadsheets:

- Compose templates consisting of mathematical formulas to perform routine arithmetic calculations for multiple data points
- Construct templates that make use of multiple step calculations and mathematical functions to perform higher-level mathematical analysis of data
- Prepare proper graphs (charts) and use them to discover trends and make predictions
- Construct calibration curves and forecast results
- Perform linear regression and other curve fitting for serial data
- Prepare publication-quality charts and tables.

7. Use electronic forms of communication:

- Use email to timely correspond with instructors and peers if situations arise
- Prepare and submit electronic pre-labs and quizzes posted on the web
- Critically evaluate web information for truth and use Internet as scientific resource
- Consult course web page for syllabus and supplemental reading assignments and other course-related materials.

8. Critically search the scientific literature for information:

- Conduct an efficient search of the scientific literature both on paper and online journals
- Use cumulative indices to locate primary literature
- Use Internet search engines to locate and evaluate scientific information
- Use journal retrieval services such as SciFinder Scholar and Google Scholar
- List and cite references in journal style reports according to ACS conventions, e.g. with EndNote.

Experimental Chemistry II - CH 464 & 464H - University Policies

Statement Regarding Students with Disabilities

Please note: "Students with documented disabilities who may need accommodations, who have any emergency medical information the instructor should know, or who need special arrangements in the event of evacuation, should make an appointment with the instructor as early as possible, no later than the first week of the term. In order to arrange alternative testing, the student should make the request at least one week in advance of

the test. Students seeking accommodations should be registered with the Office of Services for Students with Disabilities."

<http://oregonstate.edu/fa/manuals/gen/students-disabilities>

In particular, "Accommodations are collaborative efforts between students, faculty and Disability Access Services (DAS). Students with accommodations approved through DAS are responsible for contacting the faculty member in charge of the course prior to or during the first week of the term to discuss accommodations. Students who believe they are eligible for accommodations but who have not yet obtained approval through DAS should contact DAS immediately at 737-4098."

IMPORTANT: Statement of Expectations for Student Conduct

Please note: The College of Science follows the university rules on civility and honesty. These can be found at <http://oregonstate.edu/studentconduct/code/index.php>.

Cheating or plagiarism by students is subject to the disciplinary process outlined in the Student Conduct Regulations. Students are expected to be honest and ethical in their academic work. Academic dishonesty is defined as an intentional act of deception in one of the following areas:

- * **cheating**- use or attempted use of unauthorized materials, information or study aids
- * **fabrication**- falsification or invention of any information
- * **assisting**- helping another commit an act of academic dishonesty
- * **tampering**- altering or interfering with evaluation instruments and documents
- * **plagiarism**- representing the words or ideas of another person as one's own

Behaviors disruptive to the learning environment will not be tolerated and will be referred to the Office of Student Conduct and Community Standards for disciplinary actions.

“The goal of Oregon State University is to provide students with the knowledge, skill and wisdom they need to contribute to society. Our rules are formulated to guarantee each student's freedom to learn and to protect the fundamental rights of others. People must treat each other with dignity and respect in order for scholarship to thrive. Behaviors that are disruptive to teaching and learning will not be tolerated, and will be referred to the Student Conduct Program for disciplinary action. Behaviors that create a hostile, offensive or intimidating environment based on gender, race, ethnicity, color, religion, age, disability, marital status or sexual orientation will be referred to the Affirmative Action Office.”