

CHEM 5530 Spring 2018: Advanced Synthesis Lab

Professor Leo Liu

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Office Hours: Appointment via email

Meeting Time/Place: M/R 2:30 pm – 6:20 pm, Widtsoe 113 (and Maeser Lab 370, if specified)

Text: None; all materials are available via Canvas prior to lab sessions. Each student is to purchase a *bound notebook (not spiral bound)* for use as a laboratory notebook.

Lab Fee: \$75 to cover expenses of chemicals and supplies for the experiments. We have been reminded to provide lab fee information on the syllabi of lab courses that includes the dollar amount (\$75 in our case for all labs) and a sentence summarizing what the fee goes for. Our lab fees go toward equipment and supplies, as well as access time on instrumentation, and a small fraction toward Teaching Assistant support. The only exception is CHEM 5680 which has no contribution for TA support.

Course Objectives/ Goals:

Students will conduct experiments to synthesize a variety of organic and inorganic compounds applied in electrochemistry and catalysis. Instruments such as NMR, UV-visible absorption spectroscopy, infrared spectroscopy, and cyclic voltammetry (CV) are important tools in chemistry and will be used in this class.

Course Design:

I will be utilizing Canvas management system for Chem 5530. Laboratory descriptions and other materials will be available only via Canvas.

Learning Objectives:

1. Know how to record the experimental data.
2. Know how to monitor the progress of reactions.
3. Know how to carry out purification.
4. Know how to design a proper workup procedure.
5. Know how to report the experimental result.
6. Know how to find the related references.
7. Know how to collect and interpret the spectroscopic data (NMR, UV, and CV etc.) to support the experimental results.

Grading: A total of 960 points are distributed as follows:

12 Prelab questions @ 10 pts:	120 pts
12 Lab Cleanliness and Performance:	120 pts
24 lab notebook checks @ 5 pts:	120 pts
6 lab reports @ 100 pts:	600 pts
Total	965 pts

Tentative Grading Scale

(Brackets could be lowered but not raised):

A-/A	90 – 100%
B-/B/B+	80 – 89%
C-/C/C+	70 – 79%
D/D+	60 – 69%

Lab Cleanliness and Performance (120 pts): It is expected of you to be fully prepared for the experiment the day of; your performance and cleanliness of the lab will be assessed. Therefore, prior to coming to lab each week you should read the experiment description, carefully analyze the experiments to be performed, and make sure that you understand the chemistry. Along with good performance and proper lab safety techniques, there will be a lab cleanliness assessment worth a combined 10 points per week. *If the student is found to be unprepared and cannot work safely, points will be deducted and/or they may be asked to leave.* Cleanliness details will be given in a separate packet.

Prelab Questions: The prelab questions will be developed from the laboratory description and posted on Canvas to download. Carefully analyze the experiments to be performed, and make sure that you understand the chemistry. Turn in a typed copy of the answers to the questions at the beginning of lab *prior to the experiment*. Reactions schemes should be drawn by ChemDraw. Software is available for free through USU, or can be found in the science computer labs. A demonstration of operation will be shown in class.

Notebook Checks (120 pts): At the end of each lab period, you must have your notebook reviewed and signed-off by Dr. Liu or Jared Moss for clarity and completeness. A possible total of 10 points will be awarded for each week. You are expected to keep a clear notebook for each laboratory including at least the following items:

1. Table of Contents (continually updated)
2. Title
3. Synopsis (1-3 sentences) (include any notable potential safety hazards)
4. Reactions clearly written and balanced, if there are clear chemical reactions involved
5. Description of procedure including amounts of reagents used (do not write before lab)
6. Detailed observations and comments **IN YOUR OWN WORDS**.
7. Results, i.e., percent yield, percent error, etc.

At the end of the semester you should have a notebook that includes a table of contents and page numbers. This will be evaluated in the final notebook check.

Required Lab Report Format (see model report on Canvas) (600 pts):

1. Typed and emailed (jaredmosslovesJesus@gmail.com), 2-3 pages (not including attached spectra, supporting information)
2. Abstract (50 words maximum) (10%)
3. Introduction with stated purpose of experiment (20%)
4. Experimental Outline: Reaction(s) carried out, apparatus sketch(es), experimental details (20%)
5. Discussion of Results and Conclusions (50%)
6. Attached electronic copies of spectra, raw data, etc. (required as part of results above)
7. Spectra and data must be clearly labeled and documented as referenced in Results and Discussion sections. Data file name format for NMR and CV: “*date_compound_name*”, e.g. 160110_1H-FcNCl_Jared Moss; 160112_CV-MV_Jared Moss

*Refer to JACS publication style for further questions.

Chem 5530 Course Schedule (subject to change)

Date	Experiment title	Tasks
1/11 R	Course Introduction	Check-in
1/16 T*	Exp 1: Synthesis of ionic ferrocene compounds	Synthesis of FcNCl
1/18 R		Synthesis FcN(TFSI)
1/22 M		Synthesis continued
1/25 R		Characterization
1/29 M		Synthesis of [MV]Cl ₂
2/01 R	Exp 2: Synthesis of ionic methyl viologen compounds	Synthesis of [MV](TFSI) ₂
2/05 M		Synthesis continued
2/08 R		Characterization
2/12 M	Exp 3: Synthesis of highly charged viologen compounds	Synthesis of [N ₄ V]Br ₄
2/15 R		Synthesis of [N ₄ V]Cl ₄
2/20 T*		Synthesis continued
2/22 R		Characterization
2/26 M	Exp 4: Synthesis of conjugated ferrocene and pyridinium	Synthesis of pyridinium
3/01 R		Coupling of ferrocene
Break 3/5-9		
3/12 M		Synthesis continued
3/15 R		Characterization
3/19 M	Exp 5: Synthesis of a water soluble anthraquinone	Synthesis of anthraquinone
3/22 R		Synthesis of anthraquinone
3/26 M		Synthesis continued
3/29 R		Characterization
4/02 M	Exp 6: Synthesis of a tetradentate ligand and its Ni complex	Synthesis of the ligand
4/05 R		Synthesis of the Ni complex
4/09 M		Synthesis continued
4/12 R		Characterization
4/19	Lab Check-out	Fill out Check-Out sheet
4/19 R	Last lab report due	Finish all assignments

* = Monday holiday; Tuesday lab schedule

Work Schedule and Final Due Dates:

1. Students will work in pairs and conduct experiments at the course meeting time.
2. Each group will design their own work schedule for each lab period based on the time of the synthetic progress. This is best done via email with your partner.
3. Office hours with teacher and TA are scheduled by appointment.
4. Lab report due *one week after the end* of the corresponding experiment.
5. Date for final lab cleanup: Thursday, April 19, 2:30 p.m.
6. Last report due: April 19 by 5 p.m.

Laboratory Safety Requirements for Chem 5530:

Please follow the general rules and requirements listed on lab safety documentation which can be downloaded from the course Canvas. For each experiment, specific safety concerns will be emphasized.

Withdrawal Policy and 'I' Grade Policy: The administration of Chem 5530 will adhere strictly to the academic regulations stipulated in the most recent Schedule of Classes and the USU General Catalog. Withdrawal from the course will follow official USU procedures. Students are required to complete all courses for which they are registered by the end of the semester. In some cases, a student may be unable to complete all of the coursework because of extenuating circumstances, but not due to poor performance or to retain financial aid. The term 'extenuating' circumstances includes: (1) incapacitating illness which prevents a student from attending classes for a minimum period of two weeks, (2) a death in the immediate family, (3) financial responsibilities requiring a student to alter a work schedule to secure employment, (4) change in work schedule as required by an employer, or (5) other emergencies deemed appropriate by the instructor.

University Standards of Academic Integrity – “The Honor System”: Each student has the right and duty to pursue his or her academic experience free of dishonesty. The Honor System is designed to establish the higher level of conduct expected and require of all Utah State University students. This includes, but not limited to, copying text from a partner’s lab report into your own.

The Honor Pledge. To enhance the learning environment at Utah State University and to develop student academic integrity, each student agrees to the following Honor Pledge: “I pledge, on my honor, to conduct myself with the foremost level of academic integrity.” A student who lives by the Honor Pledge is a student who does more than not cheat, falsify, or plagiarize. A student who lives by the Honor Pledge espouses academic integrity as an underlying and essential principle of the Utah State University community; understands that each act of academic dishonesty devalues every degree that is awarded by this institution; and is a welcomed and valued member of Utah State University.

Grievance Process (Student Code): Students who feel they have been unfairly treated (in matters other than (i) discipline or (ii) admission, residency, employment, traffic, and parking – which are addressed by procedures separate and independent from the Student Code) may file a grievance through the channels and procedures described in the Student Code.

Plagiarism: Plagiarism includes knowingly "representing, by paraphrase or direct quotation, the published or unpublished work of another person as one's own in any academic exercise or activity without full and clear acknowledgment. I also included the unacknowledged used of

materials prepared by another person or agency engaged in the selling of term papers or other academic materials." The penalties for plagiarism are severe. They include warning or reprimand, grade adjustment, probation, suspension, expulsion, withholding of transcripts, denial or revocation of degrees and referral to psychological counseling. This includes, but not limited to, copying text from a partner's lab report into your own.

Sexual Harassment: Sexual harassment is defined by the Affirmative Action/Equal Employment Opportunity Commission as any "unwelcome sexual advances, requests for sexual favors, and other verbal or physical conduct of a sexual nature." If you feel that you are a victim of sexual harassment, you may talk to or file a complaint with the Affirmative Action/Equal Employment Opportunity Office located in Old Main, Room 161, or call the AA/EEO Office at 797-1266.

Students with Disabilities: The Americans with Disabilities Act states: "Reasonable accommodation will be provided for all persons with disabilities in order to ensure equal participation within the program." If a student has a disability that will likely require some accommodation by the instructor, the student must contact the instructor and document the disability through the Disability Resource Center (797-2444), preferably during the first week of the course. Any request for special consideration relating to attendance, pedagogy, taking of examinations, etc., must be discussed with and approved by the instructor. In cooperation with the Disability Resource Center, course materials can be provided in alternative format, large print, audio, diskette, or Braille.