

Chem 5530 (Spring 2022)
Advanced Synthesis Laboratory

Instructor: Dr. Gang Li
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Teaching Assistant: Keyang Wu
Office: ML351
Office Hours: By appointment
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Class Time: Mon/Thu 2:30 pm - 6:20 pm
Meeting Place: WIDT113 (and ML 370, if specified)

Lab Fee: The laboratory fee of **\$100** is used to repair and replace equipment, purchase chemicals, supplies, and to support teaching assistants for this course.

Course Content:

The purpose of this lab course is to help you apply your knowledge in chemical theories into applications. You will conduct experiments to synthesize a variety of organic and inorganic compounds applied in chemical catalysis. You will also learn how to use instrumentations and technics including NMR, UV-vis spectroscopy, infrared spectroscopy, recrystallization, thin-layer column (TLC), flash column, etc. to facilitate your projects.

Required Text: None (Materials will be made available via Canvas)

Recommended Resources:

- a) Scifinder: scifinder.cas.org (Most powerful search engine for chemistry. Only available on USU campus network, use VPN when off-campus)
- b) Youtube: www.youtube.com (you can find lab training videos including recrystallization, TLC, flash column on YouTube)

Laboratory Safety Requirements:

General Safety Requirement: Please follow the rules and requirements listed on the departmental website for teaching lab at: <http://www.chem.usu.edu/safety/SafetyNew-working>

COVID-19 Guidelines: Please follow the university guidelines on COVID-19 at: <https://www.usu.edu/covid-19/>

Learning Objects:

1. Know how to record the experimental data.
2. Know how to monitor the progress of reactions.
3. Know how to carry out column chromatography 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-vis, IR, *etc.*)

Grading: A total of 500 points is possible. Points are distributed as follows:

Lab Quizzes (5 × 10 pts)	50 pts
Lab Reports (5 × 50 pts)	200 pts
Lab Notebook Check (5 × 30 pts)	150 pts
Lab Cleanliness and Performance (5 × 20 pts)	100 pts

Tentative Grading Scale:

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

Scales could be lowered based on overall class performance but will **not** be **raised**.

Lab Quizzes: The lab 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 (or the time specified on the quiz). 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.

Lab Notebook Check: At the end of each lab period, you must have your notebook reviewed and signed-off by Dr. Li or Keyang Wu for clarity and completeness. A total score for each topic will be 30 points. 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.

Required Lab Report Format (see model report on Canvas) (250 pts):

1. Typed and email to Keyang Wu, 2-3 pages (not including attached spectra, supporting information). **Lab report is due one week after the completion of the experiment.**
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: Name Initial-Notebook Number (Roman Numerals)-Page Number-Types of experiments. For example, a proton NMR data on page 12 of Keyang Wu's second notebook: KW-II-12-H.

A Template for reporting NMR spectroscopic data:

^1H NMR (600 MHz, CDCl_3) δ 9.33 (s, 1H), 7.87 (dd, $J = 5.5, 3.1$ Hz, 2H), 7.76 (dd, $J = 5.5, 3.0$ Hz, 2H), 5.01 (dd, $J = 11.2, 5.2$ Hz, 1H), 2.34 – 2.20 (m, 1H), 1.83 (ddd, $J = 14.2, 9.4, 5.3$ Hz, 1H), 1.46 (q, $J = 7.8, 7.0$ Hz, 1H), 0.93 (dd, $J = 6.7, 2.3$ Hz, 6H); ^{13}C NMR (150 MHz, CDCl_3) δ 168.1, 167.8, 134.5, 131.4, 123.7, 51.4, 37.5, 25.0, 22.9, 21.3.

Please refer to JACS publication style for further questions.

Academic Freedom and Professional Responsibilities

Academic freedom is the right to teach, study, discuss, investigate, discover, create, and publish freely. Academic freedom protects the rights of faculty members in teaching and of students in learning. Freedom in research is fundamental to the advancement of truth. Faculty members are entitled to full freedom in teaching, research, and creative activities, subject to the limitations imposed by professional responsibility. [USU Policy 403](#) further defines academic freedom and professional responsibilities.

Academic Integrity - "The Honor System"

The University expects that students and faculty alike maintain the highest standards of academic honesty. The Code of Policies and Procedures for Students at Utah State University ([Student Conduct](#)) addresses academic integrity and honesty and notes the following:

Academic Integrity: Students have a responsibility to promote academic integrity at the University by not participating in or facilitating others' participation in any act of academic dishonesty and by reporting all violations or suspected violations of the Academic Integrity Standard to their instructors.

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".

Violations of the Academic Integrity Standard (academic violations) include, but are not limited to cheating, falsification, and plagiarism

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. It also includes the unacknowledged use 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.

Course Fees

Instructors that utilize course fees should identify the amount and explain the purpose of the course fee on the syllabus. Course fees are listed in the catalog.

Grievance Process

Students who feel they have been unfairly treated [in matters other than discipline, 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: [Article VII Grievances](#)

Students with Disabilities

USU welcomes students with disabilities. If you have, or suspect you may have, a physical, mental health, or learning disability that may require accommodations in this course, please contact the Disability Resource Center (DRC) as early in the semester as possible (University Inn # 101, 435-797-2444, drc@usu.edu). All disability related accommodations must be approved by the DRC. Once approved, the DRC will coordinate with faculty to provide accommodations.

Withdrawal Policy, "I" Grade Policy and Dropping Courses

If a student does not attend a class during the first week of the term or by the second class meeting, whichever comes first, the instructor may submit a request to have the student dropped from the course. (This does not remove responsibility from the student to drop courses which they do not plan to attend.) Students who are dropped from courses will be notified by the Registrar's Office through their preferred e-mail account.

Students may drop courses without notation on the permanent record through the first 20 percent of the class. If a student drops a course following the first 20 percent of the class, a W will be permanently affixed to the student's record (check [General Catalog](#) for exact dates).

Students with extenuating circumstances should refer to the policy regarding Complete Withdrawal from the University and the Incomplete (I) Grade policy in the General Catalog.

Course Schedule

Date	Topic	Tasks
01/10	Course Introduction	Lab Check-in
01/13	Exp 1: Synthesis of Hydroxamic Acids	Synthesis, purification, and characterization of hydroxamic acids
01/17	No Class (MLK Day)	
01/20	Exp 1: Synthesis of Hydroxamic Acids	
01/24	Exp 1: Synthesis of Hydroxamic Acids	
01/27	Exp 1: Synthesis of Hydroxamic Acids	
01/31	Exp 1: Synthesis of Hydroxamic Acids	
02/03	Exp 1: Synthesis of Hydroxamic Acids	
02/07	Exp 2: Synthesis of 3-substituted-1,4,2-dioxazol-5-ones	Synthesis, purification, and characterization of 3-substituted-1,4,2-dioxazol-5-ones
02/10	Exp 2: Synthesis of 3-substituted-1,4,2-dioxazol-5-ones	
02/14	Exp 2: Synthesis of 3-substituted-1,4,2-dioxazol-5-ones	
02/17	Exp 2: Synthesis of 3-substituted-1,4,2-dioxazol-5-ones	
02/21	No Class (President's Day)	
02/24	Exp 3: Synthesis of Fe Catalysts	Synthesis and characterization of Fe Catalysts
02/28	Exp 3: Synthesis of Fe Catalysts	
03/03	Exp 3: Synthesis of Fe Catalysts	
03/07	No Class (Spring Break)	
03/10	No Class (Spring Break)	
03/14	Exp 3: Synthesis of Fe Catalysts	
03/17	Exp 4: Catalytic Reaction 1: Cyclization	Conduct and evaluate the efficiency of catalyst; obtain desired product; evaluate the substrate scope
03/21	No Class (ACS Meeting)	
03/24	Exp 4: Catalytic Reaction 1: Cyclization	
03/28	Exp 4: Catalytic Reaction 1: Cyclization	
03/31	Exp 4: Catalytic Reaction 1: Cyclization	
04/04	Exp 5: Catalytic Reaction 2: C-H Activation	Conduct and evaluate the efficiency of catalyst; obtain desired product; evaluate the substrate scope
04/07	Exp 5: Catalytic Reaction 2: C-H Activation	
04/11	Exp 5: Catalytic Reaction 2: C-H Activation	
04/14	Exp 5: Catalytic Reaction 2: C-H Activation	
04/18	Reserved for Unfinished Tasks	
04/21	Summary	Lab Check-Out
04/25	Final Report Due	