

CHEM 410: ADVANCED SYNTHESIS AND CHARACTERIZATION

Spring 2018 Syllabus

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CREDIT HOURS: 3 (1 hr lecture, 6 hr lab)

PREREQUISITES: CHEM 401 (Inorganic Chemistry) or equivalent

COURSE MEETING TIMES: LECTURE, Fulmer 225: Fri 2:10 - 3:00 pm
LAB, Fulmer 445: SECTION 1 Wed, Thu 2:10 - 5:00 pm
SECTION 2 Tu, Thu 9:10-11:50 am

COURSE WEBSITE: The course website and gradebook will be maintained in Blackboard
(<http://learn.wsu.edu/>)

REQUIRED TEXT: Synthesis and Technique in Inorganic Chemistry, 3rd Ed. by Girolami, Rauchfuss and Angelici (ISBN: 978-0935702484)
(eBook: eISBN 978-1938787089)

RECOMMENDED RESOURCES: These texts listed below may provide useful explanations, but are not required for the course.

You are expected to search and read reference literature (journals and textbooks). That means using the library and SciFinder Scholar. Below is a list of textbooks you may find useful as general references. These books are available at Owen Science and Engineering Library.

1. "The Manipulation of Air Sensitive Compounds" by D. F. Shriver and M. A. Drezdon (ISBN 978-0471867739)
2. "Microscale Inorganic Chemistry" by Z. Szafran, R. M. Pike, and M. M. Singh. (ISBN 978-0471619963)
3. "The Organic Chem Lab Survival Manual: A Student's Guide to Techniques" by Zubrick (ISBN 978-1118875780)
4. "A Microscale Approach to Organic Laboratory Technique" by Pavia, Kriz, Lampman, and Engel (ISBN 978-1133106524)
5. "Prudent Practices in the Laboratory" (free download at <https://www.nap.edu/catalog/12654/prudent-practices-in-the-laboratory-handling-and-management-of-chemical>) (ISBN 978-0-309-13864-2)
6. ACS Style Guide (<http://pubs.acs.org/series/styleguide>)

LEARNING GOALS, COURSE OBJECTIVES:

Chemistry 410 is designed to advance students toward the WSU Learning Goals, especially Scientific Literacy, Critical and Creative Thinking, Quantitative Reasoning, Communication, and Information Literacy. The goals of Chem 410 are to provide students with laboratory instruction in synthesis, purification, and characterization of organometallic, inorganic, and materials compounds, as well as in writing a formal laboratory report. Chem 410 focuses on modern synthetic technology, contemporary characterization techniques, and laboratory technique development. The laboratory work is complemented with lectures highlighting pertinent synthetic techniques, specific characterization methods, and safety. Students will use

gram-scale techniques to synthesize compounds and employ a variety of characterization tools including UV-visible spectroscopy, IR (and techniques within), NMR, magnetic susceptibility, cyclic voltammetry, mass spectrometry, X-ray diffraction, and chromatographic methods. This course will reinforce the techniques and skills that synthetic chemists employ in their work and students will be encouraged to develop their lab skills in an independent manner.

By the end of the course it is expected that every student will:

- Acquire and improve their skills in the strategy of chemical synthesis.
- Work safely with air-sensitive and malodorous reagents
- Acquire and improve skills in the use of different characterization techniques and in the interpretation of material characterization data.
- Improve their speed and efficiency in the laboratory.
- Improve their ability to be self-critical (that is, to notice inconsistencies or abnormalities in their experimental findings and to take steps to check their reliability).
- Enhance their confidence in their ability to work in the laboratory.
- Enhance their ability to work safely and independently in the laboratory.
- Enhance their ability to work collaboratively in the laboratory.
- Improve their formal technical writing skills.

Each experiment can be considered as a mini research project. Since your time in the lab will be limited, you must challenge yourself to be well prepared and organized. Your success in this course depends for the most part on good preparation, good planning, good laboratory technique, and good attitude. Welcome to the real research world!

Student Learning Outcomes At the end of this course, students should be able to:	Course Topics/Dates The following topic(s)/dates(s) will address this outcome:	Evaluation of Outcome: This outcome will be evaluated primarily by:
<ul style="list-style-type: none"> • Acquire and improve their skills in the strategy of chemical synthesis. • Work safely with air-sensitive and malodorous reagents • Acquire and improve skills in the use of different characterization techniques and in the interpretation of material characterization data. • Improve their speed and efficiency in the laboratory. • Improve their ability to be self-critical (that is, to notice inconsistencies or abnormalities in their experimental findings and to take steps to check their reliability). • Enhance their confidence in their ability to work in the laboratory. • Enhance their ability to work safely and independently in the laboratory. • Enhance their ability to work collaboratively in the laboratory. • Improve their formal technical writing skills. 	Laboratory Experiments 1-11	Laboratory Reports for Experiments 1-11

COURSE CONTENT: Students will perform 11 experiments in organometallic, inorganic, and materials synthesis. These experiments were selected from *Organic Synthesis*, *Inorganic Synthesis*, the laboratory manual, and the current literature.

Tentative Course Schedule:

Dates	Laboratory Experiment
January 9 or 10, 2018	Safety Discussion
January 11, 2018	1) Glow Sticks (also Safety Quiz first 30 min of class)
January 16 or 17, 2018	2) Silicone Polymers
January 18, 2018	2) Silicone Polymers
January 23 or 24, 2018	3) Synthesis of $\text{tBuNH}_2\text{BH}_3$
January 25, 2018	3) Synthesis of $\text{tBuNH}_2\text{BH}_3$
January 30 or 31, 2018	4) Synthesis of Thermochromic Compounds
February 1, 2018	4) Synthesis of Thermochromic Compounds
February 6 or 7, 2018	4) Synthesis of Thermochromic Compounds
February 8, 2018	5) Synthesis of $\text{Mn}(\text{acac})_3$
February 13 or 14, 2018	6) Sublimation of Ferrocene
February 15, 2018	6) Synthesis of Ferrocenium PF_6
February 20 or 21, 2018	7) Synthesis of KBar^{F}_4
February 22, 2018	7) Synthesis of KBar^{F}_4
February 27 or 28, 2018	8) Synthesis of $[\text{Bar}^{\text{F}}_4]^-$ Salts (Ferrocenium Bar^{F}_4)
March 1, 2018	8) Synthesis of $[\text{Bar}^{\text{F}}_4]^-$ Salts ($\text{H}(\text{OEt})_2\text{Bar}^{\text{F}}_4$)
March 6 or 7, 2018	9) Synthesis of Salen Ligands
March 8, 2018	9) Synthesis of $\text{Co}(\text{salen})$
March 12-16, 2018	Spring Break (No Class)
March 19-23, 2018	Spring ACS (No Class)
March 27 or 28, 2018	9) O_2 Uptake with $\text{Co}(\text{salen})$
March 29, 2018	10) Synthesis of H_2TPP
April 3 or 4, 2018	10) Synthesis of $\text{Cu}(\text{TPP})$
April 5, 2018	10) Synthesis of $\text{Cu}(\text{TPP})$
April 10 or 11, 2018	11) Synthesis and Substitution of $\text{Fe}_2(\text{edt})(\text{CO})_6$
April 12, 2018	11) Synthesis and Substitution of $\text{Fe}_2(\text{edt})(\text{CO})_6$
April 17 or 18, 2018	11) Synthesis and Substitution of $\text{Fe}_2(\text{edt})(\text{CO})_6$
April 19, 2018	11) Synthesis and Substitution of $\text{Fe}_2(\text{edt})(\text{CO})_6$
April 23-27, 2018	Dead Week (No Class)
April 30 – May 4, 2018	Finals Week (No Class)

Experiments 2, 5, and 10 can be found in the laboratory manual, the remaining procedures can be found on Blackboard.

LAB REPORTS: Each student is required to use a lab notebook with numbered duplicate pages. In this notebook, each student will keep a record of collected data and experimental observation. Although you will be working in pairs, you are not permitted to use the same notebook or replicate lab reports. The laboratory reports are to be written in the style of a journal article (see the *Journal of the American Chemical Society*, *JACS*, for reference). A template for the lab report can be found on Blackboard. The reports must be written in a word processor and printed. A report is due for each completed experiment and will be turned in to supervising faculty **no later than one week after the scheduled experiment ends** on a Friday. Late lab reports will be penalized 15% per calendar day. Questions regarding a lab report or a completed experiment are welcomed and encouraged, but **not the last day** they are due. To learn how to write formal lab reports each student will be responsible for writing a single section of a lab report each week. The particular section will change from week to week and are assigned according to the table seen on page 5. In addition to writing a single section each student is required to critique a

complete lab report containing the sections that each of the students have written combined into one document. Section 1 will read and evaluate Section 2's combined lab report and Section 2 will read and evaluate Section 1's combined lab report. **The evaluation of the combined lab report will be due the following week and will count for 20 points.** All chemical structures should be drawn using ChemDraw.

Lab Report Sections:

- (a) **Title:** Title of your experiment.
- (b) **Author and Affiliation:** Your name and that of your lab partner in parenthesis. Your affiliation is Department of Chemistry, Washington State University, Pullman, WA 99164-4630.
- (c) **Abstract:** A brief summary of what was done and what was found. The abstract should contain only facts and not conclusions. It should include the most important discoveries or data and results. Limit your abstract to one paragraph.
- (d) **Introduction:** This section should contain background information about the compound being made, its scientific and practical importance, as well as relevant applications.
- (e) **Experimental Section:** A brief outline of the steps carried out in performing an experiment. Include information such as experimental observations and sketches of experimental apparatus when appropriate.
- (f) **Results and Discussion:** The section should include any calculations, graphs, spectra etc... Detailed data interpretation and analysis and discussion of experimental results should be included. Discussion of problematic areas and sources of error as well as error calculations should also be contained in this part of the lab report.
- (g) **Conclusions:** This section should contain comments on the significance, utilization, etc., of the experiment performed.
- (h) **Answers to Questions:** You may be assigned a list questions for a particular experiment. Answers to these questions must be addressed directly in this section but can also be reiterated in the Results and Discussion section, if needed.
- (i) **Supporting Information:** This part is to be taken from notes taken while working in the laboratory and is appended to your lab report. All hard data such as weight and volume readings, tabulated spectral and instrumental data, a record of significant observations such as color changes and temperature and time of reaction, extra calculations, etc., should be included in this section. A carbon copy of your notebook should simply be included as part of your lab report - it needs no further work. This section requires a cover page that contains a table of contents with page numbers of the data included in the supporting information.
- (j) **References:** This section should contain a listing of literature sources used in preparing the lab report. For examples on how to reference data found in books and journal articles consult recent JACS journal and the ACS Style Guide.
- (k) **Table of Contents Graphic:** This graphic should capture the reader's attention and, in conjunction with the manuscript title, should give the reader a quick visual impression of the essence of the manuscript without providing specific results.

For Experiments 1-10: A complete lab report will consist of the following sections: Title, Author and Affiliation, Selected Section (see table of page 5 for assigned section), Answers to Questions, a Supporting Information Section, and a Table of Contents Graphic.

The lab report for Experiment 11 will contain all of the sections a-k (on page 4).

Due Date	Experiment	Lab Report Section Assignment				
		Abstract	Introduction	Experimental	Results & Discussion	Conclusions
1/19/18	1	1,6,11	2,7,12	3,8,13	4,9,14	5,10,15
1/26/18	2	5,10,15	1,6,11	2,7,12	3,8,13	4,9,14
2/2/18	3	4,9,14	5,10,15	1,6,11	2,7,12	3,8,13
2/16/18	4	3,8,13	4,9,14	5,10,15	1,6,11	2,7,12
2/16/18	5	2,7,12	3,8,13	4,9,14	5,10,15	1,6,11
2/23/18	6	1,6,11	2,7,12	3,8,13	4,9,14	5,10,15
3/2/18	7	5,10,15	1,6,11	2,7,12	3,8,13	4,9,14
3/9/18	8	4,9,14	5,10,15	1,6,11	2,7,12	3,8,13
4/6/18	9	3,8,13	4,9,14	5,10,15	1,6,11	2,7,12
4/13/18	10	2,7,12	3,8,13	4,9,14	5,10,15	1,6,11
4/27/18	11	Full Lab Report by All Students				

GRADING SCALE AND POLICY: The grading scale will be administered in accordance to the policies of the WSU university registrar. There will be no exams given in this course. The student's final grade will be based on lab performance: experimental write-ups, experimental accuracy, and laboratory technique. Note your class participation grade will be affected negatively by the lack of preparedness for labs and the lack of attendance.

1 Formal Lab Report	100 points
10 Lab Report Sections	500 points
10 Lab Report Reviews	200 points
Safety Quiz	100 points
Class participation/Lab Technique	100 points
Course Total	1000 points

Course Grading Scale: A (1000-920), A- (919.9-900), B+ (899.9-830), B (829.9-820), B- (819.9-800), C+ (799.9-730), C (729.9-720), C- (719.9-700), D+ (699.9-630), D (629.9-620), F (619.9-0)

ONLINE CONTENT: Information of the current and upcoming experiments, course lectures, announcements, and literature will be available on the Blackboard course page (<https://learn.wsu.edu/>) for CHEM 410.

ACADEMIC INTEGRITY: Cheating, plagiarism, or any other activity, which results in an unfair advantage, will not be tolerated. Students repeating this course must rework and rewrite all lab reports. Submitting previously graded work, even if your own, is considered cheating. Cooperative learning is encouraged, but all work submitted for grading must be your own according to WSU policy found at <http://www.wsulibs.wsu.edu/plagiarism/main.html>. Identically worded lab reports are considered cheating. All instances of cheating will be reported to Student Affairs with the assignment in question receiving no credit. Repeated offenses will result in a failing grade for the course in accordance to WSU conduct policy found at <http://www.conduct.wsu.edu/default.asp?PageID=343>.

ACCOMMODATIONS: Reasonable accommodations are available for students who have a documented disability. Please notify the instructor during the first week of class of any accommodations needed for the course. Late notification may cause the requested accommodations to be unavailable. All accommodations must be approved through the Disability Resource Center (DRC) in Administration Annex 205, 335-1566 in Pullman. If you have a disability and may need accommodations to fully participate in this class, please visit the Disability Resource Center (DRC). All accommodations MUST be approved through the DRC (Admin Annex Bldg, Room

205). Please stop by or call 509-335-3417 to make an appointment with a disability specialist. If you have any questions, please contact Rosie Pavlov at pavlovr@wsu.edu or 335-3417 or check on our website at www.drc.wsu.edu

UNEXPECTED ABSENCES: Attendance is **mandatory** in this course. If you plan to be absent from the laboratory because of unforeseen or foreseen circumstances you **must** notify the instructors in writing explaining your situation, prior to the event. You must also give a copy of the letter to the TA. In addition, you need to arrange to make up the missed laboratory.

SAFETY ON CAMPUS: WSU has developed resources for the safety of students, faculty, staff and visitors. These are the Campus Safety Plan at <http://safetyplan.wsu.edu> and the university emergency management at <http://oem.wsu.edu/emergencies>. You should also become familiar with the WSU ALERT site at <http://alert.wsu.edu> for information about emergencies affecting WSU. It is recommended that you go to the myWSU portal at <http://my.wsu.edu> and register your contact information for the Crisis Communication System (CCS).

SAFETY IN LABORATORY: *You will not be permitted to begin an experiment without having read the safety instructions completely.*

Goggles are required at all times. A pair of rubber gloves is strongly recommended. Any experiments utilizing or generating a noxious or offensive gas must be done in the hood. Hg is a cumulative poison. Report any spills at once.

No one is allowed to work alone in an unsupervised laboratory. Some experiments in this course are dangerous if not carried out properly. If you need extra time to complete an experiment, see your instructor for arranging extra time.

Experiments have been selected and designed to utilize all the safety precautions and equipment available to limit chemical exposure. For additional information on safety please read the subsequent section on Teaching Lab Safety Procedures.

CHEM 410
Teaching Lab Safety Procedures

A. 1st day of lab. What to locate?

1. Note locations of fire extinguishers, both in lab and in adjacent hallway.
2. Note locations of eyewash and deluge shower facilities and fire blanket (if available).
3. Locate first aid kit.
4. Locate nearest telephone. Know emergency numbers for fire and ambulance, in our case 911.
5. Locate nearest fire alarm.
6. Note primary and secondary routes of egress from the building.

B. 1st day of lab. What to know?

1. That you are responsible for the safety and welfare of yourself and of those working around you.
2. What to do in event of fire.
 - a. Get an extinguisher and put it out.
 - b. If you cannot put it out:
 - i. If you are using a gas burner, turn the gas off.
 - ii. Leave the room. Make sure everyone else does too.
 - iii. Close the door.
 - iv. Leave the building. When leaving do not waste time, (picking up belongs, putting away equipment, etc.). Someone should meet the fire fighters on the South side of the building, (toward Physical Sciences), to direct them to the fire. -- Never use an elevator to escape a fire.
 - c. Your laboratory instructor will demonstrate the proper use of fire extinguishers, safety showers, and eyewash facilities.

C. Prudent Laboratory Practices.

1. Personal protective equipment.
 - a. Approved eye protection is mandatory.
 - i. Protection from splashes of solvents and corrosives.
 - ii. Protection from impact hazards.
 - iii. Goggles are available at the chem club goggle sale and at the Bookie. NOTE: Any eye injury, no matter how slight, requires immediate medical attention
 - b. Lab coats are required for Chem 410. They are available at the Bookie.
 - c. Wear gloves when handling toxic or corrosive chemicals. Disposable gloves are available in the lab.
 - d. Shoes are mandatory. No sandals or open toes are allowed.
 - e. Make use of the fume hoods.
2. General Practices
 - a. Confine long hair.
 - b. Do not eat, drink, smoke, or taste anything in the lab.
 - c. Use due caution when using open flames.
 - d. Never perform unauthorized or unsupervised experiments.
 - e. Get medical attention for any injuries, other than the most minor cuts and burns, incurred in the lab. Whenever you do so, notify your instructor and have him or her help you to fill out an accident report.
 - f. Report failures of laboratory utilities or equipment to your instructor.

D. Safety with Chemicals.

1. Before using a given chemical know the following:
 - a. The hazards associated with its use.
 - b. The means of protection from its hazards.
 - c. What to do in case of spill or other emergency.
 - d. How to properly dispose of it (Prudent Practices in the Laboratory is a good resource).
2. The above knowledge can be obtained from the following:
 - a. Your text and/or laboratory manual.
 - b. The label of the chemical container.
 - c. Your instructor.

The following is a general discourse on hazards and procedures associated with chemicals as categorized by hazard class, (i.e., flammable, corrosive and toxic).

Note that there is a great deal of variation within hazard classes and that many chemicals belong to more than one hazard class.

Occasionally, a chemical will require special cautions or procedures. Your instructor will notify you when such a chemical is introduced to the lab.

E. Flammables. Examples; acetone, ether, methanol.

1. Fire hazard. Vapors form an ignitable mixture with air. If an ignition source, (flame or spark), is present the vapor/air mixture will ignite and can flash back to the vapor source.

Most flammable vapors will be **heavier** than air and will therefore sink to low levels.

Most of the flammable solvents are immiscible with water, thus water is impractical for controlling solvent fires.

F. Protection from lab fires.

1. Keep flammables stored in designated storage areas and containers.
2. Eliminate ignition sources such as open flames, hotplates, and electrical equipment.
3. Handle solvent containers carefully.
4. Be conservative in the amounts of flammables you use or transfer. Don't take more than is necessary to get the job done.
5. Keep flammables isolated from other reactive chemicals (oxidizers, reducers, acids, bases).
6. Transfer and use flammables under fume hoods.

G. In case of a flammable liquid spill.

1. Remove any potential ignition sources.
2. Notify your instructor.
3. The instructor or his/her designee will go to the storeroom for spill pillows. Each pillow will absorb about one liter of liquid.
4. Use the pillow to absorb the spill. Place the pillow in a plastic bag. Close the bag securely and tag it with the name of the spilled chemical. Take the bag to the storeroom for disposal.

H. In case an individual, (you or someone near you) catches fire to his/her person or clothing:

1. **Stay calm.**
2. Walk, don't run, to the nearest safety shower and extinguish the flames.
3. Notify your instructor.

I. Disposal of waste flammables.

1. Put wastes in the container designated for Waste Flammables Only. Be careful not to mix with other waste classes.
2. Mark the name and amount of the chemical you are disposing of on the list on or adjacent to the waste container.
3. Keep the outside of the waste container and the area around it clean.
4. If you have any questions about waste disposal, ask your instructor.

J. Corrosives (usually acids and bases). Example: Sulfuric acid, hydrochloric acid, ammonium hydroxide.

- 1. Hazards:** Corrosives act on body tissues through direct contact with the skin and eyes and via inhalation or ingestion.

Corrosive injury can range from irritation to physical disruption of body tissues.

Note that in some cases knowledge of contact and subsequent corrosive injury may be delayed until the action is advanced.

2. Protection from corrosives:

- a. Goggles.
- b. Gloves.
- c. Lab Coat. Protects both you and your clothes.
- d. Shoes.
- e. Fume hood.

3. In case of a corrosive chemical spill.

- a. Notify your instructor.
- b. Depending on the size and type of the spill it may be flushed or diluted with water and/or may be picked up with a spill pillow. Your instructor will decide.

4. In case of contact with a corrosive chemical:

- a. Immediate flushing of the affected area with water is critical.
Use an eyewash station, safety shower, or sink as appropriate to the situation.
Flush the affected area for 15 minutes. While flushing remove and discard any clothing which may be contaminated with the corrosive.
- b. Notify your instructor.
- c. The product label should be consulted for first aid information. If medical attention is warranted provide label information to the attending medical personnel. Remember, any eye injury must receive immediate medical attention.
- d. Neutralizers and solvents other than water should not be used in first aid treatment.

5. Disposal of waste corrosives.

- a. Most of the mineral acids and common bases can be flushed down the sink. Slowly and carefully flush them down with plenty of cold water.
- b. Your instructor will notify you of any corrosives which must be collected in waste containers.
- c. In most cases it is important to keep corrosive wastes segregated from other classes of wastes. Addition of highly reactive chemicals to the wrong waste container can result in the release of highly toxic fumes, fire or explosion.
- d. Ask your instructor if you are unsure of the proper disposal procedure.

K. Toxic chemicals (Poisons)

1. Hazards

- a. Toxicity can be classified as acute or chronic.
Acute toxicity is based on short term, high dosage exposure. Chronic toxicity is based on long term, low dosage exposure. Symptoms, injurious effects, and relative severity of affects of acute intoxication may be quite different from those of chronic intoxication by the same chemical.
- b. Routes of exposure to toxicants include:
 - a. Absorption through the skin or eyes.
 - b. Inhalation
 - c. Oral ingestion
 - d. Penetration by mechanical injury with contaminated glass or metal .

2. Protection from toxic chemicals.

- a. Gloves
- b. Goggles
- c. Lab coat
- d. Fume hood.
- e. Use of good lab practices.
- f. Good personal hygiene.

3. In case of toxic chemical spill.

- a. Notify your instructor.
- b. Spill will be cleaned up according to previously outlined procedures.

4. In case of contact with a toxic chemical:

- a. Notify your instructor.
- b. Follow first aid instructions on the product label.
- c. In case medical attention is required give label information to attending personnel.

L. Disposal of Waste toxic chemicals.

1. Put wastes in container designated for Toxic Wastes Only. Be careful not to mix with other waste classes.
2. Mark the name and amount of the chemical you are disposing of on the list on or adjacent to the waste container.
3. Keep the outside of the waste container and the area around it clean.
4. If you have any questions about waste disposal ask your instructor.

Chemistry 410 Lab Map (Fulmer 445): Please add the safety equipment to study for the safety quiz on January 11th.

