CHEM 410: ADVANCED SYNTHESIS AND CHARACTERIZATION

Spring 2015 Syllabus

INSTRUCTORS: Prof. Paul Benny, email: bennyp@wsu.edu, phone: 509-335-3858
Office hours: by appointment

LABORATORY ASSISTANTS: Mr. Tom Hayes, email: trhayes@wsu.edu
Office hours: by appointment

Ms. Shalina Bottorff, email: shalina.bottorff@email.wsu.edu
Office hours: by appointment

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

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

LEARNING GOALS, COURSE OBJECTIVES:

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 as part of the Writing in the Major Course (M-Course) requirement. 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, transmission electron microscopy, electron microscopy, scanning probe microscopy, 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. Specific learning goals are to:

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

The laboratory reports are to be written is the style of a journal article (see the *Journal of the American Chemical Society, JACS*, for reference). Students will receive a formal review of their report from the instructor and will have the opportunity to make appropriate corrections to their work. Resubmitted reports will evaluated for final grading.

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!

COURSE CONTENT: Students will perform 5 experiments in organometallic, inorganic, and materials synthesis. These experiments were selected from *Organic Synthesis*, *Inorganic Synthesis*, and current literature. These experiments are:

1. Purification and characterization of bis(cyclopentadienyl)iron (II), ferrocene. Synthesis and characterization of ferrocene carboxylic acid.
2. Decarbonylation and complexation of Mn carbonyl complexes.
3. Synthesis and characterization of bis(diethylammonium)tetrachlorocuprate(II); demonstration of thermochromism.
4. Preparation of enzyme mimics of bioinorganic Fe dehydrogenase complexes.
5. Preparation of an explosive: Nitrogen triiodide ammoniate.

A schedule of experiments and lectures is given on separate pages. You will be provided with a copy of each experiment at the appropriate time by the instructor. The experimental procedures will not be extensively detailed. They will be similar to the procedures found in the literature. We encourage you to provide creative input and learn to think and act independently in the laboratory.

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

1. "The manipulation of Air Sensitive Compounds" by D. F. Shriver and M. A. Drezdon.
2. "Advanced Practical Organic Chemistry" by M. Casey, J. Leonard, B. Lygo, G. Procter.
3. "Vogel's Textbook of Practical Organic Chemistry" by B. S. Furniss, A. J. Hannaford, P. W. G. Smith, and A. R. Tatchell.
4. "Synthesis and Techniques in Inorganic Synthesis" 2nd Ed. by R. J. Angelici.
5. "Handbook of Preparative Inorganic Chemistry" vol. 1 and 2 by G. Brauer.
6. "Microscale Inorganic Chemistry" by Z. Szafran, R. M. Pike, and M. M. Singh.
7. ACS Style Guide.

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 lab reports are to be written is the style of a journal article. The *Journal of the American Chemical Society* publications will serve as our model. The reports must be word processor 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. Questions regarding a lab report or a completed experiment are welcomed and encouraged but **not the last day** they are due. Reports will be formally reviewed and returned to the students for corrections. Students will be allowed to resubmit their corrected reports for final grading no later than **1 week** after receiving their reviews form the instructor.

Each lab report must include the following 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**: This segment should not be done in your notebook. 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 may be incorporated into the Results and Discussion section or addressed directly in this section.

(i) **Associated Content**: This part is to be taken from notes taken while working in the laboratory and is a separate document from your lab report manuscript. 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.

(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.

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.

4 Formal Lab reports	400 points
Class participation	50 points
Course Total	**450 points**
Course Grading Scale:	A (450-405), A^- (405-396), B^+ (395-387), B (386-360), B^- (359-351), C^+ (350-342), C (341-315), C^- (314-306), D^+ (305-297), D (296-270), F (269-0)

ONLINE CONTENT: Information of the current and upcoming experiments, course lectures, announcements, and literature will be available on the Angel course page for CHEM 410 at www.lms.wsu.edu.

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 TAs. 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 9ll.
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 PLU goggle sale and at the Bookie. NOTE: Any eye injury, no matter how slight, requires <u>immediate</u> 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.

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. <u>Immediate</u> 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.