CHEMISTRY 4:153 (CHEM:3530:AAA) **Fall 2014**
INORGANIC SYNTHESIS LABORATORY

Prof. Lou Messerle
Office: E435 Chemistry Building (CB); **Phone**: 335-1372
Course email: chem-course-messerle@uiowa.edu (please place "Chem 4:153 Inorganic Lab" in title)
Graduate Teaching Assistants: Justine Olson, Nicholas Schnicker, Madeline Basile
Pre-Laboratory Lecture: Monday and Wednesday, 11:30 AM-12:20 PM, CB W268
Laboratory Location, Times Section **A01**: Tuesday, Thursday, 2:00-4:50 PM, CB E424
Section **A02**: Monday, Wednesday, 2:30-5:20 PM, CB E424
(rarely, a lab may be extended ≤ 30 min in order to reach optimal experiment stopping point)
Course credit: Three credit hours: lab reports, homework, midterm exam, final and lab practical exam
Office hours: held in CB E427 to be determined after survey of students's available times
<u>Course Website (under development):</u> 004:153:AAA Fall14 Inorganic Chemistry Laboratory, Iowa Courses Online (ICON) website URL = http://icon.uiowa.edu/. Use your HawkID and HawkID password to log in to ICON. Lecture notes, practice exams, course announcements, exam announcements and room assignments, exam keys, and other info will be posted on ICON, so the instructors encourage you to check ICON frequently.
Textbook: Any good practical laboratory techniques manual, such as those used in organic lab courses 4:141/142; handouts will be provided for all experiments
Course Reserves: Several inorganic lab books and text books will be on reserve in Science Library Annex (CB W223; Monday-Thursday 9:30-noon) and/or available electronically. For techniques:

1. S. Komiya, "Synthesis of Organometallic Compounds: A Practical Guide" (eReference)
2. G. Girolami, T. Rauchfuss, R. Angelici, "Synthesis and Technique in Inorganic Chemistry: A Laboratory Manual"
3. R. Errington, "Advanced Practical Inorganic and Metalorganic Chemistry"
4. W. Jolly, "The Synthesis and Characterization of Inorganic Compounds"
5. J. Tanaka, S. Suib, "Experimental Methods in Inorganic Chemistry"
6. Z. Szafran, R. Pike, M. Singh, "Microscale Inorganic Chemistry: A Comprehensive Laboratory Experience"

Course Description

Synthetic chemistry represents the beginning of the science of chemistry and is undisputably Chemistry's foundation. Pure compounds are used in a wide variety of practical applications, from materials to pharmaceuticals, and in research in a wide variety of sciences. Important compounds are often unavailable commercially (or too costly) and need to be synthesized, purified, and characterized. Theories about the properties, uses, structures, and reactivities of unknown molecules can only be tested by first synthesizing a previously unreported target molecule, and such syntheses and their reaction products test and expand our knowledge and theories about chemical bonding.

Inorganic chemistry is the oldest chemical science and is the foundation for many materials that form the basis for our technological society, from metals and alloys such as bronze (first discovered millenia ago) to iron and steel, efforts to transmute base metals to gold by the alchemists, uranium in the nuclear age, and solid-state materials and devices based on silicon. From its practical origins in metallurgy, inorganic chemistry has exploded over the last 60 years to cover bioinorganic, organometallic, and materials chemistries. Part of the reason is the considerable landscape, only explored in miniscule detail, of the chemistry of the multitude of elements beyond carbon and their countless combinatorial permutations.

This laboratory course is designed to teach students advanced synthetic chemistry laboratory techniques, complementary to those learned in 4:141/142, for the preparation, purification, and characterization of inorganic, organic, and air-sensitive organometallic molecules and materials via macroscale and microscale methods. Approaches to searching and finding literature procedures for compound preparation will be reviewed. This course will emphasize inorganic and organometallic compounds and materials of the lanthanide and transition elements, but many of the techniques are applicable to organic synthesis. Particular course emphases include

1. developing student confidence in designing and safely executing molecular syntheses at macro and micro synthetic scales, solving real-life problems along the way,
2. taking students beyond the cookbook approaches of earlier lab courses in the sciences, and
3. developing hands-on, practical (as opposed to abstract physical principles in other courses) experience in modern spectroscopic characterization techniques, especially high field multinuclear FT NMR spectroscopy. New last year was an experiment in microscale synthesis using microscale apparatus similar to that used in pharmaceutical and radiochemical synthesis, and this year a new experiment relevant to solar energy conversion will be introduced.

Pre-laboratory lectures will include discussion of the upcoming experiments (including safety aspects required for safe execution of the experiment), bench and spectroscopic techniques used in the experiments, demonstration of related topics in synthesis and characterization, and discussion of the results. The midterm exam will be given during one lecture period in October.

Several multi-step synthetic experiments in contemporary inorganic chemistry and a session on laboratory glassblowing (glassblowing knowledge/skills are often needed in organometallic chemistry, as well as in reactions in bioinorganic and materials chemistries) are planned for the semester. Portions of the last experiment will involve collaborative research with chemists at Caltech in their NSF-funded Center for Chemical Innovation, CCI Solar, and may result in a paper to be submitted to a journal with contributing students' names as coauthors. The tentative lecture, lab experiment, and exam schedule (subject to change as new experiments are designed and older experiments re-evaluated) are shown on the last page.

Lecture topics to be covered may include: recrystallization, inert-atmosphere compound manipulations by Schlenk line/glove box/glove bag techniques, Soxhlet extraction, sublimation, mechanical stirring, rotary evaporation, vacuum pump and trap utilization, practical FT NMR spectroscopy, practical IR spectroscopy, practical mass spectrometry, interpretation of IR/NMR/mass spectra, introduction to powder and single-crystal X-ray diffractometry, tube and muffle furnace use in high-temperature solid-state inorganic synthesis, polarimetry, and (possibly) magnetochemistry.

Materials to be purchased by student: lab notebook (inexpensive bound notebook or composition book, with pages numbered by student), protective rubber gloves, and comfortable goggles or safety glasses with splash shields.

Miscellaneous

Please feel free to discuss with Prof. Messerle any aspect of the course that is of concern or causing you problems. DON'T HESITATE to come to office hours to ask questions that are not adequately covered, from your perspective, during class. If you require course adaptations or accomodation because of a recognized disability, contact Prof. Messerle who will work to accomodate your needs.

Course Administration

Please go to the Chemistry Center, E225 CB, for drop/add signatures. M–F, 8:00 AM-12:00, 1:00-5:00 PM (F, 4:30 PM). Manager: Rudy Marcelino (335-1341, rudy-marcelino@uiowa.edu).

Grading

The overall grade will be based on laboratory reports and the exam grades, with the laboratory reports constituting the major portion of the grade. An approximate breakdown is:

Homework/pre-lab assignments	50 points
Midterm exam	100 points
Final exam (includes lab practical exam)	200 points
Cobalt coordination chemistry	90 points
High T_C superconductor synthesis	90 points
Glassblowing	20 points (no report)
Tungsten cluster chemistry	90 points
Supramolecular chemistry/X-ray diffraction	90 points
Tungsten organometallic chemistry	90 points
Dirhenium quadruple bond/microscale	90 points
Solar fuels-relevant chemistry	90 points
	1000 points total

Laboratory reports are due on the schedule date, generally one week after experiment completion; 10 points deducted for **each day** that the report is late. The reports should be closely modeled after the format used for full articles in the Journal of the American Chemical Society (please consult a recent issue), with the following sections: Abstract; Introduction; Experimental Section; Results; Discussion; References. The laboratory report should be concise (≤ 8 pages typed or 16 pages hand written; no credit for illegible lab reports; appendix pages do not count in total) and should include spectra and copies of the lab notebook pages in an appendix. Each report will be graded according to the following criteria:

Evidence of thought	30 points
Quantitative results	15 points
Treatment of data	15 points
Performance in laboratory	20 points
Organization of report and laboratory notebook	10 points
TOTAL	90 points

1. Evidence of thought
   The Abstract should be a single paragraph explaining the basic purpose(s) and result(s) of the experiment. The Introduction should elaborate on the basic purpose of the experiment, with relevant literature references, and give balanced chemical equations. The Experimental Section should discuss techniques, list reagents used in the experiment, and include a matter flowchart, a concise way for demonstrating your understanding of the experiment. Flowcharting is a method for showing the logical flow of the steps and compounds/by-products/solvents in the experiment, and as such it is preferable to a simple listing (i.e., copying) of the experimental procedure from the laboratory handout. The Discussion section should answer questions posed in the handout for each experiment, detail your ideas on how to improve yields and the experiment in general, give the stoichiometry of the principal and side reactions in the experiment with balanced equations, and demonstrate that you understood the purpose(s) of the experiment.
   We are looking for evidence that you did more than simply "cookbook" the experiment.

2. Quantitative results
   The Results section should give percentage yield and data concerning purity and characterization of any compound prepared. All other data and observations should be provided.

3. Treatment of data
   The experimental data must be analyzed in the Discussion section in terms of error analysis (if applicable) and use of significant figures. Sample calculations should be shown, and graphs supplied when requested or needed for analysis.

4. Performance in laboratory
   This portion of the grade is determined by the TAs; it is based on their evaluation of the degree of preparation that the student demonstrates during execution of the experiment, understanding of the techniques employed in the experiment, and organization of time spent in the laboratory. Knowledge and use of appropriate safety precautions will be especially noted, in particular the wearing of goggles whenever the student is in the lab. Grade reductions for repeated failure to observe safety precautions will be used in the grading of the report, in addition to the possible banning of the student from the laboratory and the resulting forfeit of credit for the experiment.

5. Report/notebook organization
   The lab report should be patterned after the format used for full articles in the Journal of the American Chemical Society (JACS). Please go to the Library website and, under eJournals, select this journal and review a synthesis-related full paper (not a Communication to the Editor). Things that are required in the report should be easily found and properly organized, and all data should be present in the Results section. Copies of lab notebook pages must be appended to the report. The proper use of a laboratory notebook is the mark of a good experimentalist (and chemistry is, after all, an experimental science). The notebook need not be a work of art but must be legible. It is NOT necessary or desirable to recopy your data/observations in the lab notebook from the day's work AFTER the lab period, and it is NOT necessary/desirable to set it up for filling in data BEFORE the lab period. The laboratory notebook should be liberally covered with observations and raw data, drawings of apparatus, and your ideas and/or thinking during the course of the experiment, in addition to the inevitable water stains from your neighbor's astray condenser lines.

Safety

Students must comply always with lab safety rules for their personal safety and the safety of others. Students must complete lab safety training and pass a quiz before they will be allowed to perform experiments. If a student fails to comply with safety rules, the student will be asked to leave the laboratory and their grade will be lowered. While in the laboratory, you must wear safety goggles or other positive eye protection at all times. During your first laboratory period, locate the positions of the fire extinguishers, showers, face sprays/eye washes, and fire blanket. Be certain that you know how to use them. Water-cooled equipment such as condensers that must operate unattended between lab periods **must be set up in fume hoods and also must have the water hoses secured with metal clips** (supplied by us) in order to prevent room flooding and water damage below. All organic solvents are assumed to be flammable and to have some degree of toxicity. Waste solvents and reagents are to be disposed of in accordance with TA instructions. For safety reasons, you are to work in the lab only during your scheduled lab period. Missed labs cannot be made up, and you should not arbitrarily choose to cut (miss) lab. Arrangements for use of instruments outside of the regular lab periods may be made with the instructors.

Pregnancy: Many chemicals pose potential hazards to a fetus or young child. Women who are pregnant, nursing, or who expect to become pregnant are strongly advised to consult with their physician about the hazards of possible exposure to chemicals used in this course. Material safety data sheets (MSDS) and other safety information are available.

Complaints

Complaints and appeals regarding the course and instructor can be filed with the Departmental Executive Officer (DEO, Prof. Sarah Larsen) at the Department of Chemistry administrative office, Room E331 CB (335-1350). Students are encouraged to meet first with Prof. Messerle (no impact on your grade) with their concerns about course aspects, lectures, or assignments.

Helpful Hints (to help you get the most out of the course and to earn a good grade)

1. Make efficient use of your time in the lab! Reading your experiment beforehand and answering pre-lab questions will help you plan your work for the next laboratory period. Know exactly what the experiment requires and estimate how long each step will take based on the experimental procedures. Certain reactions require several hours to go to completion. Begin these first, so that you can work on other parts of the experiment while those reactions are proceeding.
2. When handing in products from your preparations, remember that a smaller amount of pure product is generally better than a large amount of contaminated, "dirty" product.
3. Your reports should be scientific papers, not novels. Write exactly what you mean, no more and no less. Avoid verbose and flowing descriptions without omitting essential information. Make it easy for the reader to determine exactly what you did, how they could reproduce it, and your results.
4. Your instructors are here for the sole purpose of teaching you more advanced synthetic techniques than you were exposed to in Chemistry 4:141/142, in addition to laboratory safety. **Do not hesitate to ask questions** when you are unsure of some aspect of the experiment, even if you think that your question is "stupid"; no question is stupid, especially involving a question about safety, and will not impact your grade. **Don't hesitate** to bring questions to Prof. Messerle's office hours or to arranged alternate times. Synthesis is a major aspect of the chemical sciences, and we want to ensure that you develop proper laboratory techniques for future research and technical work in either graduate school or industrial/government employment.

Course-Specific Academic Honesty (in addition to CLAS policies on next page)

Examinations: You are expected to work alone. Out of fairness for all students, cheating will not be tolerated. The instructors believe strongly in fairness for all students and objective appraisal of individual performance and understanding of material.

Laboratory: An experiment and its data collection may occasionally occur as a small group activity. All data must be collected in the lab. Use of data not collected by the lab report's author or partner, data not acquired during lab period, and/or fabricated data are serious academic misconduct. We encourage you to discuss technique and lab questions in groups, but questions in the lab report must be answered individually by you.

College of Liberal Arts and Sciences (CLAS): Additional Policies and Procedures

Administrative Home: CLAS is the administrative home of 4:153 and governs matters such as add/drop deadlines, second-grade-only option, and other related issues. Different colleges may have different policies. Questions may be addressed to 120 Schaeffer Hall, or see the CLAS Academic Policies Handbook: http://clas.uiowa.edu/students/handbook/.

Electronic Communication: University policy specifies that students are responsible for all official correspondences sent to their University of Iowa e-mail address (@uiowa.edu). Students should check their account frequently and use this account for course correspondence. The course instructors and TAs will typically try to respond to student e-mails within 2 academic days (M-F).

Academic Honesty: All CLAS students have, in essence, agreed to the College's Code of Academic Honesty (http://clas.uiowa.edu/students/handbook/academic-fraud-honor-code): "I pledge to do my own academic work and to excel to the best of my abilities, upholding the IOWA Challenge (http://thechallenge.uiowa.edu): I promise not to lie about my academic work, to cheat, or to steal the words or ideas of others; nor will I help fellow students to violate the Code of Academic Honesty." Any student committing academic misconduct is reported to the College, placed on disciplinary probation, and/or may be suspended or expelled from the University.

CLAS Final Examination Policies: The final examination schedule for each class is announced around the fifth week of the semester by the Registrar. Final exams are offered only during the official final examination period. **No exams of any kind are allowed during the last week of classes.** All students should plan on being at the UI through the final examination period, so don't plan family vacations or travel during this period. The schedule will be published on the Registrar's web site

Understanding Sexual Harassment: Sexual harassment subverts the mission of the University and threatens the well-being of students, faculty, and staff. All members of the UI community have a responsibility to uphold this mission and to contribute to a safe environment that enhances learning. Incidents of sexual harassment should be reported immediately. See the UI Comprehensive Guide on Sexual Harassment at http://www.uiowa.edu/~eod/policies/sexual-harassment-guide/index.html for assistance, definitions, and the full University policy.

Reacting Safely to Severe Weather: In severe weather, students should seek appropriate shelter immediately, leaving the classroom if necessary. The class will continue if possible when the event is over. For more information on Hawk Alert and the siren warning system, visit the Public Safety web site: http://police.uiowa.edu/stay-informed/emergency-communication/.

Resources for students:

1) Writing Center: 110 English-Philosophy Building (EPB), 335-0188, www.uiowa.edu/~writingc
2) Speaking Center: 412 EPB, 335-0205, clas.uiowa.edu/rhetoric/for-students/speaking-center
3) Mathematics Tutorial Laboratory: 125 MacLean Hall, 335-0810, www.math.uiowa.edu/MathTutorialLab/index.shtml
4) Tutor Iowa: tutor.uiowa.edu
5) College of Engineering Tutoring Program: www.engineering.uiowa.edu/sdc/tutoring.php
6) Supplemental Instruction: tutor.uiowa.edu/supplemental-instructions/
7) University Housing Tutoring: housing.uiowa.edu/departments/reslife/academic_initiatives.html

University Examination Policies

Missed exam: UI policy requires that students be permitted to make up exams missed because of illness, religious obligations, certain University activities, or unavoidable circumstances.

Final Examinations: A student with two final examinations scheduled for the same period or more than three examinations on the same day may file a request for a change of schedule before the published deadline at the Registrar's Service Center, 17 Calvin Hall, 8-4:30 M-F (384-4300).

(VERY) TENTATIVE LABORATORY AND ASSIGNMENTS SCHEDULE **8/27 v**

A02 SECTION DAY/DATE	A01 SECTION DAY/DATE	EXPERIMENT	LECTURE TOPIC(S)	ASSIGN-MENT DUE DATE
M Aug 25	Tu Aug 26	Check-in, safety training, instrumentation tour	Synthesis strategies; lab notebook; $Co(en)_3^{3+}$, enantio/diastereomers	
W Aug 27	Th Aug 28	$Co(en)_3^{3+}$ synthesis	Recrystallization, polarimetry	
M Sept 1	Tu Sept 2	**NO LAB – Labor Day**		
W Sept 3	Th Sept 4	$Co(en)_3^{3+}$ synthesis, resolution	SciFinder review	
M Sept 8	Tu Sept 9	$Co(en)_3^{3+}$ synthesis, racemization	Superconductivity	
W Sept 10	Th Sept 11	$YBa_2Cu_3O_{7-\delta}$ superconductor: sol-gel synthesis	Superconductivity: Meissner effect	
M Sept 15	Tu Sept 16	$YBa_2Cu_3O_{7-\delta}$: sol-gel synthesis of student-concocted material; muffle furnace		
W Sept 17	Th Sept 18	$YBa_2Cu_3O_{7-\delta}$ superconductor: fly-off	Glassblowing	
M Sept 22	Tu Sept 23	Glassblowing: torch; cutting, flaring, firepolishing tubing	Glassblowing	
W Sept 24	Th Sept 25	Glassblowing: bending, joining tubing, T-joint and butt seal	Air-sensitive technique: overview	
M Sept 29	Tu Sept 30	Glassblowing: making a bubbler	Metal clusters	
W Oct 1	Th Oct 2	$W_6Cl_{14}^{2-}$ synthesis: glovebag, seal ampule	Schlenk line	
M Oct 6	Tu Oct 7	$W_6Cl_{14}^{2-}$ isolate, purify acid salt	Schlenk line	
W Oct 8	Th Oct 9	$W_6Cl_{14}^{2-}$ isolation of Bu_4N^+ salt	FT NMR	
M Oct 13	Tu Oct 14	$W_6Cl_{14}^{2-}$ characterization	Supramolecular and green chemistry; XRD	
W Oct 15	Th Oct 16	Supramolecular chemistry	FT NMR	
M Oct 20	Tu Oct 21	Supramolecular chemistry	Midterm Exam???	
W Oct 22	Th Oct 23	Supramolecular chemistry	Organotransition metal chemistry	
M Oct 27	Tu Oct 28	Organoditungsten chemistry	FT NMR	
W Oct 29	Th Oct 30	Organoditungsten chemistry	IR; MS	
M Nov 3	Tu Nov 4	Organoditungsten chemistry	Dinuclear chemistry; M-M multiple bonding; microscale technique	
W Nov 5	Th Nov 6	Microscale dirhenium metal-metal multiple bond synthesis		
M Nov 10	Tu Nov 11	Microscale Re=Re		
W Nov 12	Th Nov 13	Microscale Re=Re	Solar energy conversion	
M Nov 17	Tu Nov 18	Solar fuel chemistry		
W Nov 19	Th Nov 20	Solar fuel chemistry	Excited state photochemistry	
	Th Nov 27	Characterize cranberry sauce		
M Dec 1	Tu Dec 2	Solar fuel chemistry	Glove box technique	
W Dec 3	Th Dec 4	Solar fuel chemistry	Spectrofluorimetry	
M Dec 8	Tu Dec 9	Solar fuel chemistry		
W Dec 10	Th Dec 11	**CHECK-OUT**		