CHEMISTRY 4:153, INORGANIC SYNTHESIS LABORATORY Fall 2007

Prof. Lou Messerle Office: CB 381 lou-messerle@uiowa.edu 335-1372

Office hours: Tuesday 10:00-11:00; Wednesday 10:30-11:20; or by appointment
Teaching Assistants: David Rotsch, Adil Mohammad (office hours to be announced)
Pre-laboratory lecture: Tuesday and Thursday @ 1:30 PM in 134 Blank Honors Center
Laboratory location and times: Tuesday and Thursday, 2:30-5:20 PM, CB 211E
Course web page on ICON: http://icon.uiowa.edu/
Textbook: Any good practical laboratory techniques manual, such as those used in organic lab courses 4:141/142; xerox handouts will be provided for all experiments, and will be posted on ICON course web site
Materials to be provided by student: lab notebook (no need for an expensive, fancy lab notebook such as those sold in local bookstores; inexpensive bound notebook or composition book, with pages numbered by student), protective rubber gloves, comfortable goggles or safety glasses with splash shields on top and sides, paper towels, small bottle of dishwashing detergent

Course Philosophy, Organization, and Content

Synthetic chemistry represents the intellectual beginnings of the science of chemistry and is its intellectual core and foundation. Pure known and new compounds are used in a wide variety of applications, from materials to pharmaceuticals, and in research. Many known compounds are often unavailable commercially and therefore must be synthesized, purified, and characterized/analyzed for purity. New compounds based on biomolecular modelling studies, other calculations such as enzyme docking studies or pharmacogenomics, or theories that predict a novel molecular property or potential application, must be prepared, 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. Such syntheses and their products test and expand our knowledge and theories about chemical bonding, and improved synthetic routes enable future research.

Inorganic chemistry itself is the oldest chemical science and is the foundation for many materials that formed the basis for ancient civilization and now for our technological society, from metals and alloys such as bronze (discovered millenia ago), to iron and steel, alchemical efforts to transmute base metals to gold, uranium in the Atomic Age, and solid-state electronic materials and devices based on silicon. From its intellectual beginnings in metallurgy, inorganic chemistry has exploded over the last 50 years to cover bioinorganic, catalysis, coordination, materials, and organometallic chemistries. Part of the reason for this is the considerable landscape, only explored in miniscule detail, of the chemistry of the multitude of elements beyond carbon and their nearly infinite combinatorial and compositional permutations.

This laboratory course is designed to teach the student advanced synthetic chemistry laboratory techniques, complementary to those learned in 4:141/142, for the preparation, purification, and spectroscopic and spectrometric characterization of inorganic, organic, and organometallic molecules and materials. Methods for searching the literature to find procedures for compound preparation will also be reviewed. The course will emphasize inorganic and organometallic compounds and materials of the main group, 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 various 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 the physical principles emphasized in other courses) experience in modern spectroscopic characterization techniques, especially high field multinuclear FT NMR spectroscopy. New this year are experiments in microscale synthesis, using microscale apparatus similar to that used in pharmaceutical and radiochemical synthesis, and transition metal organometallic chemistry.

Prelaboratory lectures during the semester will include discussion of the week's experiments (including the safety aspects required for safe execution of the experiment), lab bench and spectroscopic techniques to be utilized in the experiments, demonstration of related topics in synthesis and characterization, and discussion of the results. Unannounced quizzes and a scheduled midterm exam will be given during this lecture time period.

Several multi-step synthetic experiments in contemporary inorganic chemistry and a session on laboratory glassblowing (glassblowing skills are commonly needed in organometallic chemistry, as well as in reactive aspects of bioinorganic and materials chemistries) are planned for the semester. Portions of two experiments involve research, and these projects could result in a paper to be submitted to a journal with contributing students' names as coauthors, as happened several years ago. The tentative lecture, lab experiment, and exam schedule, subject to change as new experiments are tested and in-lab modifications are introduced, is shown on the Course Schedule document.

Lecture topics to be covered include: recrystallization, inert-atmosphere compound manipulations by Schlenk line/glove box/glove bag techniques, methods for running reactions, solvent purification, Soxhlet extraction, sublimation, mechanical stirring, rotary evaporation, vacuum pump and trap utilization, cryogen and pressurized gas handling and safety aspects, practical CW and FT NMR spectroscopy, practical IR spectroscopy, practical mass spectrometry, interpretation of IR/NMR/mass spectra, introduction to X-ray diffractometry, tube and muffle furnace use in high temperature inorganic synthesis, and possibly magnetochemistry if time permits.

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:

Glassblowing, Chemical Abstracts search	35 points (no report)
Homework	35 points
Quizzes	30 points
Midterm exam	100 points
Final exam (includes lab practical exam)	200 points
High T_c superconductor synthesis	100 points
Coordination chemistry	100 points
Ditungsten/dirhenium quadruple bond	100 points
Gallium dibromide synthesis	100 points
Metal cluster chemistry	100 points
Tungsten NO organometallic chemistry	100 points
	1000 points total

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

Evidence of thought	30 %
Quantitative results	20 %
Treatment of data	20 %
Performance in laboratory as judged primarily by TAs	20 %
Organization of report and laboratory notebook	10 %

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 suitable literature references. The Experimental Section should discuss the techniques and list the reagents used in the experiment and should include a flowchart, which is a concise way for demonstrating your understanding of the experiment. Flowcharting is a method for showing the logical flow of the steps and compounds in the experiment, and as such it is preferable to a simple listing (i.e., copying) of the experimental procedure from the laboratory handout. A sample flowchart is given on the ICON web site. The Discussion section should detail your ideas on how to improve yields and the experiment in general, should give the stoichiometry of the principal and side reactions in the experiment with balanced equations, should demonstrate that you understood the purpose of the experiment (other than "because it's required"), and should answer any questions posed in the handout for each experiment.

In general, we are looking for evidence that you did more than simply "cookbook" the experiment.

2. Quantitative results

The Results section should of course give percentage yield and data concerning the purity and characterization of any compound prepared. All other data determined in the experiment should be listed.

3. Treatment of data

The experimental data must be properly 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 primarily by the TAs, with supplemental observations by Prof. Messerle; it is based on their evaluation of the degree of preparation that the student demonstrates during execution of the experiment, of the degree of understanding of the techniques employed in the experiment, and of the degree of 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. Demerits for repeated failure to observe safety precautions will be subtracted in the grading of the report, in addition to the possible banning in extreme cases of the offending student by Prof. Messerle from the laboratory and the resulting forfeit of credit for the experiment.

In general, we are judging whether we would be comfortable in working alongside the student in a synthesis laboratory.

5. Report/notebook organization

The lab report should, as mentioned above, be patterned after the format used for full articles in the Journal of the American Chemical Society. Students are strongly encouraged to visit the Library's electronic journals and look up several synthesis-related full papers (as opposed to Communications tot he Editor, which in general lack data and experimental sections) as models. Things that are required in the report should be easily found and properly organized, and all tabulated data should be present in the Results section. Copies of the appropriate laboratory notebook pages (either yellow carbon copies or xeroxes of the original lab notebook pages) should 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), and it is this which we will be checking. The notebook need not be a work of art, but at least legible. It is NOT necessary or desirable to recopy your data and observations from the day's work AFTER the laboratory period, and it is NOT necessary or even desirable to set it up for filling in the data BEFORE the lab period. It is my feeling that the laboratory notebook should be liberally covered with observations (especially and obviously raw data), drawings of the apparatus utilized in the experiment, 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 always comply with laboratory safety rules for their personal safety and the safety of other students and instructors. 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 glasses 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 which must operate unattended between lab periods **must be set up in fume hoods (which have recessed surfaces that direct water spills to a hood drain) and also must have the water hoses secured with metal clips** (supplied by us) to prevent accidental flooding of the room and water damage to lower floors; these water accidents sometimes occur in laboratories because of variations in water supply line pressures. All organic solvents are assumed to be flammable and to have varying degrees of toxicity. Waste solvents and reagents are to be disposed of in accordance with the TA's instructions, usually in labelled waste bottles in a fume hood. For safety reasons, you are to work in the lab only during the scheduled lab period. Missed labs in general 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 will be made available.

Laboratory Charges

After you are checked in (before the first synthetic experiment), you will be responsible for breakage of any items that you use in the course. This includes the equipment that you are issued in your drawer for the entire semester and specialized equipment which is utilized for individual experiments. All apparatus must be returned **clean** at the end of the semester, or it will be considered as "broken" and you will be charged for its replacement.

Complaints

Complaints and appeals regarding this course, its instructors, and/or its TAs can be filed with the Departmental Executive Officer (Prof. David F. Wiemer) at the Department of Chemistry administrative offices, Room 305 CB (335-1350). Students are encouraged to meet with Prof. Messerle with their concerns about course aspects, lab partners, TAs, lectures, or exams at any time; such discussions will be kept confidential at the student's request.

Helpful Hints (to help you get the best grade and, more importantly, the most out of the course)

1. Make efficient use of your time in the lab! Reading your experiment beforehand 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. 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 to the TAs the products of your preparations, remember that a smaller amount of pure material is generally better than a large amount of contaminated material.

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. In short,

make it easy for the reader to determine exactly what you did and what results you obtained. A good rule of thumb: write it in such a way that another student would understand what, how, and why you did the experiment, and what you observed. In other words, what would you need and appreciate to know in order to reproduce the work of another student?

4. Your instructors are here for the sole purpose of teaching you more advanced synthetic chemistry 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 believe that your question is a stupid one; no question is stupid, especially if involving a question about safety. **Do not hesitate** to bring questions to Prof. Messerle's scheduled office hours or to arranged alternate times.

The techniques and logic that you will learn in this course are applicable to many areas outside chemistry– for example, an understanding of the nuts-and-bolts of FT NMR spectroscopy is invaluable to an understanding of magnetic resonance imaging (MRI) in medicine, air-sensitive technique is applicable to handling disease pathogens (protecting a compound from air and moisture is remarkably similar to sterile technique for protecting you from the nasty biologics, and the biologics from you), and microscale technique is useful for handling small quantities of radioactive materials.

Chemical synthesis is a major aspect of the science of chemistry, and we want to ensure that you develop effective laboratory techniques for future research and technical work in either graduate school or industrial/government employment.

REQUIRED INFORMATION FROM COLLEGE OF LIBERAL ARTS AND SCIENCES

Academic Fraud: All forms of plagiarism and any other activities that result in a student presenting work that is not his or her own are academic fraud. All academic fraud is reported first to the Departmental DEO and then to the Associate Dean for Academic Programs and Services. See Academic Fraud at http://www.clas.uiowa.edu/students/academic_handbook/ix.shtml for the complete policy. Prof. Messerle had to deal with an academic fraud issue last semester in 4:12, and would prefer to not have to deal with another.

Making a Suggestion or a Complaint: Students have the right to make suggestions or complaints and should first visit with the instructor, then with the course supervisor if necessary, and next with the Departmental DEO. All complaints must be made as soon as possible. For more information visit, Student Complaints at http://www.clas.uiowa.edu/students/academic_handbook/ix.shtml#5

Accommodations for Disabilities: Under the Americans with Disabilities Act and Section 504 of the Rehabilitation Act of 1973, instructors must provide reasonable academic accommodations for qualified students with disabilities. Students seeking academic accommodations first register with Student Disability Services and meet with a counselor in that office who reviews documentation and determines eligibility for services. Students approved for accommodations arrange to meet privately with course instructors. Visit Student Disability Services at http://www.uiowa.edu/~sds/.

Understanding Sexual Harassment: Sexual harassment is reprehensible and will not be tolerated by the University. It subverts the mission of the University and threatens the well-being of students, faculty, and staff. Visit this site (http://www.sexualharassment.uiowa.edu/) for definitions, assistance, and the full University policy.

Recommended Policy Information

Resources for Students:

Writing Center 110 English-Philosophy Building, 335-0188, www.uiowa.edu/~writingc

Speaking Center 12 English-Philosophy Building, 335-0205, www.uiowa.edu/~rhetoric/centers/speaking

Mathematics Tutorial Laboratory 314 MacLean Hall, 335-0810, www.uiowa.edu/mathlab

Tutor Referral Service Campus Information Center, Iowa Memorial Union, 335-3055, www.imu.uiowa.edu/cic/tutor_referral_service

Student Classroom Behavior: Students have the right to a classroom environment that encourages learning. The ability to learn is lessened when students engage in inappropriate classroom behavior, distracting others; such behaviors also is a violation of the Code of Student Life. When disruptive activity occurs, a University instructor has the authority to determine classroom seating patterns and to request that a student exit the classroom, laboratory, or other area used for instruction immediately for the remainder of the period. One-day suspensions are reported to appropriate Departmental, collegiate, and Student Services personnel (Office of the Vice President for Student Services and Dean of Students).

University Examination Policy: **Final Examinations.** An undergraduate student who has two final examinations scheduled for the same period or more than three examinations scheduled for 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).

Missed exam policy. University policy requires that students be permitted to make up examinations missed because of illness, mandatory religious obligations, certain University activities, or unavoidable circumstances. Excused absence forms are required and are available at the Registrar web site: http://www.registrar.uiowa.edu/forms/absence.pdf