Title of Course: CHEMISTRY (CHEM:3530:0AAA0
INORGANIC CHEMISTRY (SYNTHESIS) LABORATORY

Course meeting times and places:
Lectures: Monday, Wednesday 11:30 AM-12:20 PM, CB E264
Laboratory: Monday, Wednesday 2:30-5:20 PM, CB E414 (section 0A02)
           Tuesday, Thursday 2:00-4:50 PM, CB E414 (section 0A01)

Department of Chemistry: https://chem.uiowa.edu/
Course ICON site: To access the course site, log into Iowa Courses Online (ICON)
https://icon.uiowa.edu/index.shtml using your Hawk ID and password.

Course Home
The College of Liberal Arts and Sciences (CLAS) is the home of this course, and CLAS
governs the add and drop deadlines, the “second-grade only” option (SGO), academic
misconduct policies, and other undergraduate policies and procedures. Other UI colleges
may have different policies.

Prof. Lou Messerle
Office location: CB E457
Student drop-in hours (conference room CB E427): Monday 4:40-5:30
       Tuesday 10:00 AM - 12:00 PM
Students are invited to drop by during these hours to discuss
questions about the course material or their concerns. Prof. Messerle is also
available by appointment if you are unable to attend his drop-in hours.
Phone: 319-331-6212 (cell)
E-mail: lou-messerle@uiowa.edu (please place CHEM:3530 in title line)
DEO: Prof. Leonard MacGillivray, Chemistry office, CB E331 (319-335-1350)

Laboratory Teaching Assistants
Jacob Schuely (Monday/Wednesday lab section)
Office and Student Drop-in Hours: CB E208, M and W 5:30-6:30 PM
   e-mail: jacob-schuely@uiowa.edu
Vidumini Samarasiri (Monday/Wednesday lab section)
Office and Student Drop-in Hours: CB E208, F 9:30-10:30 AM;
   also, virtual meeting Tu 9:30-10:30 AM, Zoom, meeting ID 399 294 2800; link;
   https://uiowa.zoom.us/j/3992942800
   e-mail: vidumini-samarasiri@uiowa.edu
Holly Huther (Tuesday/Thursday lab section)
Office and Student Drop-in Hours: CB E208, W and Th 9:30-10:30 AM
   e-mail: holly-huther@uiowa.edu
Balaka Bhuniya (Tuesday/Thursday lab section)
Office and Student Drop-in Hours: CB E208, W and F 8:30-9:30 AM
   e-mail: balaka-bhuniya@uiowa.edu
Description of Course

Chemical synthesis represents the beginning and foundation of the science of Chemistry. 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 for applications in analytical, biological, and physical chemistries. 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, especially in inorganic and organometallic chemistries.

Inorganic chemistry is the original 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 prepared millenia ago) to iron and steel, efforts to transmute base metals into gold by the alchemists (as a way to fool their leader/king/queen into funding actual research), uranium in the nuclear age, catalysts for petrochemical refining and organic synthesis, and solid-state materials and devices. From its practical origins in metallurgy, inorganic chemistry of the main group, transition metal, lanthanide, and actinide elements has exploded over the last 80+ years to cover bioinorganic, organometallic, environmental, and materials chemistries. Part of the reason for this expansion is the considerable landscape, only explored in small detail, of the chemistry of the multitude of elements beyond carbon (which is typically combined with hydrogen, oxygen, nitrogen, and other main group elements).

This laboratory course is designed to teach students safe, advanced synthetic chemistry laboratory techniques, complementary to those learned in organic laboratory courses, for the preparation, purification, and characterization of inorganic compounds and materials, air-sensitive organometallics, and compounds via macroscale and microscale methods. Approaches to searching and finding literature procedures by SciFinder for compound preparation will be reviewed. This course will emphasize inorganic and organometallic compounds and materials of the transition elements, but many of the techniques are applicable to organic synthesis (e.g., a palladium-catalyzed Suzuki coupling to make a new C-C bond for a new ligand). Particular course emphases include:

1. developing student confidence in designing and safely executing molecular syntheses at macro and micro synthetic scales, solving real-lab problems along the way,
2. taking students beyond the cookbook approaches of earlier lab courses in the sciences,
3. developing hands-on, advanced practical (as opposed to abstract physical principles in other courses) experience in modern spectroscopic characterization techniques, especially high field multinuclear 1D FT NMR spectroscopies, mass spectrometry, powder and single-crystal X-ray diffractometry, spectrofluorimetry, combustion/elemental analysis, new synthetic techniques involving Schlenk lines, glove bags, glove boxes, and microscale approaches (also relevant to pharmaceutical synthesis and radiochemistry/nuclear medicine).
4. learning the basics of borosilicate glassblowing in order to design, construct, and repair scientific glassware, and to seal under vacuum borosilicate glass ampules for compound storage or synthesis.

Pre-lab lectures will include discussion of the upcoming experiments (particularly safety aspects required for the experiment), bench and spectroscopic techniques used in the experiments, demonstration of related topics in synthesis/characterization, and discussion of results.

A number of multi-step synthetic experiments in contemporary inorganic/organometallic chemistry are planned for the semester. Several experiments are designed to give students opportunities for actual research in a CURE (Course-related Undergraduate Research Experience) fashion and allow publication of student results in the Chemistry literature (as has occurred in the past). Portions of the last experiment, if completed, will involve collaborative research with inorganic chemists at the California Institute of Technology (Caltech, where Prof. Messerle spent 9 months on a Career Development Award) and may result in a paper to be submitted to a journal with contributing students’ names as coauthors. The tentative lecture, lab experiment, due dates, and exam schedule (subject to change as new experiments are designed and older experiments re-evaluated) are shown later in this syllabus.
Lecture topics to be covered include: recrystallization, inert-atmosphere compound manipulations by Schlenk line/glove box/glove bag techniques, Soxhlet extraction, sublimation, mechanical stirring, rotary evaporation, vacuum pump and safe liquid-nitrogen 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, spectrofluorimetry, and polarimetry.

Materials to be purchased by student: lab notebook (inexpensive bound notebook/composition book, like one used in grammar school, with pages numbered by student), rubber gloves, and comfortable goggles that prevent splashes and impact from imploding evacuated glassware. A flame-resistant lab coat is highly recommended for protection of your clothing and may make you feel more “professional”.

Learning Objectives

Appended to this syllabus are general Chemistry program undergraduate educational outcomes, with some slight editing by Prof. Messerle. More specific learning outcomes for CHEM:3530 are:

Laboratory Skills:
1) Learning the logic of chemical synthesis; choosing best synthetic approach depending on defined constraint(s) (costs in materials and labor, time, availability of starting reagents, etc.)
2) Proper use and legal aspects (outside of UI) of laboratory notebooks
3) Safe handling of air- and moisture-sensitive reagents and reagent solutions by syringe, cannula, Schlenk line, glovebag, and research-grade glove box methods
4) Sol-gel synthesis of solid-state materials, sintering by muffle and tube furnaces, preparing pellets of solid-state materials by hydraulic press
5) Microscale techniques for synthesis using expensive reagents (e.g., platinum group metals) and/or for future radiochemical synthesis (e.g., nuclear medicine)
6) Advanced isolation/purification strategies including fractional recrystallization, fractional distillation, sublimation, Soxhlet extraction, and drying or removal of volatile contaminants via an Abderhalden drying pistol
7) Characterization of inorganic and organometallic compounds (and, by extension, organic compounds) by multinuclear 1D NMR spectroscopies, UV-vis spectrophotometry, mass spectrometry, infrared spectroscopy, spectrofluorimetry, elemental analysis, powder X-ray diffractometry, and single-crystal X-ray diffractometry; caveats about over-reliance on a single characterization technique
8) Preparation and use of a Grignard (organomagnesium) reagent for an organodirhenium compound

Chemical Information Skills:
1) Specific approaches for using SciFinder for finding literature syntheses and/or molecular characterization data, with caveats.
2) Learning about new types of compounds: transition metal-transition metal single and multiple bonds, transition metal clusters, high-Tc superconductors, photoreductants

Textbook/Materials

There remains no good textbook for the experiments in this course, as much of the chemistry is from the more recent literature. Any good, practical laboratory techniques manual, such as those used in organic lab courses, may be useful. Documents on techniques for experiments will be provided on ICON for all experiments.

Course Reserves

Several excellent but older inorganic lab technique books, an eReference, and textbooks with older experiments, will be on reserve in the Science Library, and possibly, for extra copies held by the Sciences Library, in CB E237 Science Library Annex, Conrad Bendixen, 319-467-1395; conrad-bendixen@uiowa.edu; Monday-Friday 8:00-5:00; waiting for confirmation). The eReference is available online through the Library web site. For most techniques:

Academic Honesty and Misconduct
All students in CLAS courses are expected to abide by the CLAS Code of Academic Honesty. Undergraduate academic misconduct must be reported by instructors to CLAS according to these procedures.

Course-Specific Academic Honesty
Examinations and Quizzes: You are expected to work alone. Out of fairness for all students, cheating and plagiarism will, simply, not be tolerated. Prof. Messerle believes strongly in fairness for all students and objective appraisal of individual performance and understanding of material.
Laboratory: All data must be collected during the lab period, unless there is a short-term use of an instrument arranged with a Teaching Assistant. Use of data not collected by the lab report’s author, data not acquired during lab period, and/or fabricated data constitute serious academic misconduct. We encourage you to discuss technique and lab questions in groups, but questions in your lab report must be answered individually by you.

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. Efficient multi-tasking is an important skill to develop in multiple-step chemical synthesis.
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 product. Please fill in the supplied labels and attach these to your vials of compounds as well as to reaction vessels in use if left for a later lab period.
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 organic chemistry laboratory courses, in addition to more advanced laboratory safety. **Do not hesitate to ask questions** when you are unsure of some aspect of the experiment, even if you fear 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 drop-in hours, arranged alternate times, teaching assistant drop-in hours, or while Prof. Messerle is in the lab room (often). Synthesis is a major aspect of the chemical sciences, and we want to help you develop proper, safe laboratory techniques for future technical work in graduate school or industrial/government/private employment.

Student Complaints
Students with a complaint about a grade or a related matter should first discuss the situation with Prof. Messerle and, if necessary, with the Chemistry DEO, Prof. Leonard MacGillivray. Students should contact CLAS Undergraduate Programs for support when the matter is not resolved at the previous levels.
Drop Deadline for this Course
You may drop an individual course before the deadline; after this deadline you will need collegiate approval. You can look up the drop deadline for this course here. When you drop a course, a “W” will appear on your transcript. The mark of “W” is a neutral mark that does not affect your GPA. Directions for adding or dropping a course and other registration changes can be found on the Registrar’s website. Students can find policies on dropping and withdrawing here.

Grading System and the Use of +/-
In this course, F grades are given only to students who represent a clear danger to themselves, nearby students, Prof. Messerle, or laboratory teaching assistants.

Course Grades
Final course grades will be assessed based on your performance in the following activities. The overall grade will be based on laboratory reports, homework, quizzes (if given), and exams, with the laboratory reports constituting the major portion of the grade. An approximate breakdown is:

<table>
<thead>
<tr>
<th>Activity</th>
<th>Points</th>
</tr>
</thead>
<tbody>
<tr>
<td>Homework (3-4 assignments)</td>
<td>37</td>
</tr>
<tr>
<td>Midterm exam (during an October class)</td>
<td>100</td>
</tr>
<tr>
<td>Final exam</td>
<td>200</td>
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<tr>
<td>Pre-lab assignments</td>
<td>63</td>
</tr>
<tr>
<td>Cobalt coordination chemistry report</td>
<td>80</td>
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<tr>
<td>Re₂ quadruple bond/microscale report</td>
<td>90</td>
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<tr>
<td>High Tc superconductor synthesis report</td>
<td>80</td>
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<tr>
<td>Dimolybdenum organometallic chemistry report</td>
<td>90</td>
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<tr>
<td>Hexatantalum cluster chemistry report</td>
<td>80</td>
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<tr>
<td>Solar fuels-relevant WL₆ chemistry report</td>
<td>100</td>
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<tr>
<td>Single-crystal XRD report</td>
<td>80</td>
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</tbody>
</table>

1000 points total

Laboratory reports are due on the scheduled date, generally one week after experiment completion; 10 points will be deducted for each day that the report is late. The reports should be closely modeled after the style for full articles in the Journal of the American Chemical Society (please consult a recent issue on Science Library journals website), with the following sections: Abstract; Introduction; Experimental Section; Results; Discussion; References. A sample lab report will be available on ICON for the first experiment. The laboratory report should be concise (≤ 8 pages printed or 16 pages handwritten; no credit for illegible lab reports; appendix pages do not count in total) and must append spectra and copies of the lab notebook pages. Each report will be graded by teaching assistants according to the following criteria:

<table>
<thead>
<tr>
<th>Criteria</th>
<th>Points</th>
</tr>
</thead>
<tbody>
<tr>
<td>Evidence of thought</td>
<td>25-30</td>
</tr>
<tr>
<td>Quantitative results</td>
<td>15</td>
</tr>
<tr>
<td>Treatment of data</td>
<td>15</td>
</tr>
<tr>
<td>Performance in laboratory</td>
<td>15-20</td>
</tr>
<tr>
<td>Organization of report and laboratory notebook</td>
<td>10</td>
</tr>
<tr>
<td>TOTAL</td>
<td>80-90</td>
</tr>
</tbody>
</table>

1. Evidence of thought
The Abstract should be a single paragraph, in your own words, 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 that may be 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 product(s)
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. This is not the “Joy of Cooking” (an old, classic gourmet cookbook) for chemists.

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 and use of significant figures (if applicable). Sample calculations and graphs should be included.

4. Performance in laboratory
   This portion of the grade is determined by your TAs; it is based on their evaluation of the degree of preparation that you demonstrated 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, face mask, and face shields and following physical distancing guidelines whenever you are in the lab. Grade reductions for repeated failure to observe safety and Covid-19 precautions will be used in the grading of the lab report, in addition to the possible banning of you from the laboratory and the resulting forfeit of credit for the experiment. This has actually happened in other lab courses.

5. Report/notebook organization
   The lab report must 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 appropriate to recopy your data/observations in the lab notebook from the day’s work AFTER the lab period, and it is NOT appropriate to set it up for filling in data BEFORE the lab period. The notebook should be liberally covered with observations (e.g., color changes), raw data, drawings of apparatus, and your ideas and/or thinking during the course of the experiment, in addition to the inevitable water stains.

Date and Time of the Final Exam
   The final examination date and time will be announced by the Registrar generally by the fifth week of classes and it will be announced on the course ICON site once it is known. Do not plan your end of the semester travel plans until the final exam schedule is made public. It is your responsibility to know the date, time, and place of the final exam. According to Registrar’s final exam policy, students have a maximum of two weeks after the announced final exam schedule to request a change if an exam conflict exists or if a student has more than two exams in one day (see the policy here).
## TENTATIVE Calendar of Lectures, Experiments, Course Assignments, and Exams

<table>
<thead>
<tr>
<th>DAY/DATE</th>
<th>DAY/DATE</th>
<th>LABORATORY COMPONENT</th>
<th>LECTURE TOPIC(S) for Monday, Wednesday @ 11:30 AM</th>
<th>ASSIGNMENT DUE DATE</th>
</tr>
</thead>
<tbody>
<tr>
<td>M Aug 22</td>
<td>Tu Aug 23</td>
<td>Check-in, safety training, lab tour</td>
<td>Safety; Co(en)$_3^{3+}$ experiment: enantiomers, diastereomers, polarimetry, racemization</td>
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<tr>
<td>W Aug 24</td>
<td>Th Aug 25</td>
<td>Co(en)$_3^{3+}$ synthesis: enantiomers</td>
<td>Synthesis strategies and criteria; lab notebook</td>
<td></td>
</tr>
<tr>
<td>M Aug 29</td>
<td>Tu Aug 30</td>
<td>Co(en)$_3^{3+}$ synthesis: resolution by forming diastereomers and separating by crystallization; polarimetry</td>
<td>SciFinder use in searching for molecular or material syntheses; Molecular isolation approaches</td>
<td></td>
</tr>
<tr>
<td>W Aug 31</td>
<td>Th Sept 1</td>
<td>Co(en)$_3^{3+}$ synthesis: conversion of least-soluble diastereomer to enantiomer; racemization; polarimetry</td>
<td>Purification methods: fractional distillation; recrystallization, how to modify approach; sublimation</td>
<td></td>
</tr>
<tr>
<td>W Sept 7</td>
<td>Th Sept 8</td>
<td>NO LABS Monday Labor Day (Sept. 5) and Tuesday (Sept. 6)</td>
<td>Glassblowing tools and glass components; cutting tubing; torch use</td>
<td>SciFinder homework due F Sept 9, 3:30 PM</td>
</tr>
<tr>
<td>M Sept 12</td>
<td>Tu Sept 13</td>
<td>Co(en)$_3^{3+}$ synthesis: finish any remaining synthesis step; polarimetry if needed</td>
<td>Glassblowing design steps</td>
<td></td>
</tr>
<tr>
<td>W Sept 14</td>
<td>Th Sept 15</td>
<td>Glassblowing</td>
<td>Molecular characterization approaches, pitfalls: How do you know you made your compound?</td>
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</tr>
<tr>
<td>M Sept 19</td>
<td>Tu Sept 20</td>
<td>Glassblowing</td>
<td>UV spectroscopy, spectrofluorimetry</td>
<td></td>
</tr>
<tr>
<td>W Sept 21</td>
<td>Th Sept 22</td>
<td>Glassblowing: making a gas bubbler</td>
<td>Metal clusters, utility, approaches to their synthesis; glove bags; Ta$_6$(µ-Br)$_2$Br$_4$/Ta$_3$(µ$_3$-Br)(µ-Br)$_3$Br$_3$(NCCH$_3$)$_3^{2+}$ experiment</td>
<td>Co(en)$_3^{3+}$ report due Friday Sept 23, 3:30 PM</td>
</tr>
<tr>
<td>M Sept 26</td>
<td>Tu Sept 27</td>
<td>Ta$_6$(µ-Br)$_2$Br$_4$/Ta$_3$(µ$_3$-Br)(µ-Br)$_3$Br$_3$(NCCH$_3$)$_3^{2+}$ cluster solid-state syntheses: practice ampule sealing; glove bag use; tube furnace</td>
<td>Solid-state synthetic approaches; superconductivity; Hi Tc perovskite superconductors; Meissner effect</td>
<td></td>
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<tr>
<td>W Sept 28</td>
<td>Th Sept 29</td>
<td>Ta$_6$(µ-Br)$_2$Br$_4$/Ta$_3$(µ$_3$-Br)(µ-Br)$_3$Br$_3$(NCCH$_3$)$_3^{2+}$ cluster isolation and recrystallization; Soxhlet extraction with metathesis to Bu$_4$N$^+$ salts</td>
<td>Practical FT NMR spectroscopy</td>
<td></td>
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<tr>
<td>M Oct  3</td>
<td>Tu Oct  4</td>
<td>( \text{Ta}_6(\mu_3-\text{Br})_2\text{Br}_4^{4+} / \text{Ta}_6(\mu_3-\text{Br})_2\text{Br}_3(\text{NCCH}_3)_2 ) cluster compound characterization (UV/vis spectroscopy, spectrofluorimetry)</td>
<td>Practical FT NMR spectroscopy</td>
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<tr>
<td>W Oct  5</td>
<td>Th Oct  6</td>
<td>( \text{YBa}_2\text{Cu}<em>3\text{O}</em>{7-\delta} ) superconductor: sol-gel synthesis; muffle furnace</td>
<td>Practical FT NMR spectroscopy</td>
<td></td>
</tr>
<tr>
<td>M Oct 10</td>
<td>Tu Oct 11</td>
<td>( \text{YBa}_2\text{Cu}<em>3\text{O}</em>{7-\delta} ) superconductor: pellet press; tube furnace</td>
<td>Air-sensitive technique: Schlenk line; iSchlenk; Schlenk techniques</td>
<td></td>
</tr>
<tr>
<td>W Oct 12</td>
<td>Th Oct 13</td>
<td>( \text{YBa}_2\text{Cu}<em>3\text{O}</em>{7-\delta} ) superconductor: Meissner effect iSchlenk</td>
<td>Organotransition metal chemistry; M-CO and M-M bonding; organodimolybdenum experiment</td>
<td></td>
</tr>
<tr>
<td>M Oct 17</td>
<td>Tu Oct 18</td>
<td>Organodimolybdenum chemistry: synthesis of (( \eta_5\text{-C}_5\text{H}_5 ))( \text{OC} )( _2 )( \text{Mo-} )( \text{Mo(CO)}_2(\eta_5\text{-C}_3\text{H}_3) ) (Mo-Mo bond)</td>
<td>Infrared spectroscopy: KBr pellets, demountable solution cells, sealed solution cells; Mass spectrometry, parent and molecular ions, fragmentation, isotope patterns for non monoisotopic elements</td>
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<tr>
<td>W Oct 19</td>
<td>Th Oct 20</td>
<td>Organodimolybdenum chemistry: isolation, recrystallization</td>
<td>MIDTERM EXAM Wednesday during lecture time</td>
<td>Y( \text{Ba}_2\text{Cu}<em>3\text{O}</em>{7-\delta} ) simple lab report due F Oct 21 at 3:30 PM</td>
</tr>
<tr>
<td>M Oct 24</td>
<td>Tu Oct 25</td>
<td>Organodimolybdenum chemistry: characterization ('H and ( ^{13}\text{C} ) NMR; IR; mass spectrometry)</td>
<td>Dirhenium experiment; M-M multiple bonding; microscale techniques</td>
<td></td>
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<tr>
<td>W Oct 26</td>
<td>Th Oct 27</td>
<td>Microscale dirhenium metal-metal quadruple bond synthesis: Re metal conversion to Re( _2\text{Cl}_6 )</td>
<td>Grignard reagent preparation; Unusual, little-studied metal-metal multiple bonds: unbridged Re( _2(\eta^1\text{-allyl})_n ), W( _2(\text{CH}_2\text{CMe}_3)_6 )</td>
<td>Organodimolybdenum report due F Oct 28 at 3:30 PM</td>
</tr>
<tr>
<td>M Oct 31</td>
<td>Tu Nov  1</td>
<td>Microscale Re( _n\text{Re} ); Grignard reagent preparation, reaction with Re( _2\text{Cl}_2(\mu_3-\text{O}_2\text{CR})_2 )</td>
<td>Solar energy conversion; Excited-state photochemistry; W(CNAr''Ar)_n experiment</td>
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<tr>
<td>W Nov  2</td>
<td>Th Nov  3</td>
<td>Microscale Re( _n\text{Re} ); new compound isolation, characterization</td>
<td>Metal-catalyzed cross-coupling chemistry in organic synthesis; further details on W(CNAr''Ar)_n experiment</td>
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<tr>
<td>Date</td>
<td>Date</td>
<td>Topic</td>
<td>Details</td>
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<tr>
<td>M Nov 7</td>
<td>Tu Nov 8</td>
<td>W(CNAr’Ar₆) (CNAr’Ar = isocyanide) solar fuel chemistry: Suzuki-Miyaura coupling to prepare ArAr’NHCHO (coupled formamide)</td>
<td>Approaches to phosphine removal from cross-coupling reactions</td>
<td></td>
</tr>
<tr>
<td>W Nov 9</td>
<td>Th Nov 10</td>
<td>W(CNAr’Ar₆): Coupling reaction workup; removal of PPh₃ with Merrifield reagent; dehydration of a supplied formamide to isocyanide by use of Burgess reagent</td>
<td>Dirhenium report due F Nov 12 at 3:30 PM</td>
<td></td>
</tr>
<tr>
<td>M Nov 14</td>
<td>Tu Nov 15</td>
<td>W(CNAr’Ar₆): Isolation and purification of isocyanide ligand by flash chromatography</td>
<td>Glove box design and techniques; safe handling of Hg and alkali metal reductants</td>
<td></td>
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<tr>
<td>W Nov 16</td>
<td>Th Nov 17</td>
<td>W(CNAr’Ar₆): reductive coupling of isocyanide with WCl₄(THF) in glove box using Na amalgam</td>
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<tr>
<td>Th Nov 24</td>
<td></td>
<td>CHARACTERIZE CRANBERRY SAUCE, MASHED POTATOES</td>
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<td></td>
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<tr>
<td>M Nov 28</td>
<td>Tu Nov 29</td>
<td>W(CNAr’Ar₆): isolation, purification by low T crystallization in glove box</td>
<td></td>
<td></td>
</tr>
<tr>
<td>W Nov 30</td>
<td>Th Dec 1</td>
<td>W(CNAr’Ar₆): characterization, elemental analysis, UV, spectrofluorimetry</td>
<td>Single-crystal X-ray diffractometry; SHELX and OLEX software</td>
<td></td>
</tr>
<tr>
<td>M Dec 5</td>
<td>Tu Dec 6</td>
<td>W(CNAr’Ar₆)SCXRD, single-crystal solid-state molecular structure determination of a student’s crystalline W(CNAr’Ar₆)</td>
<td>W(CNAr’Ar₆) report due TBD</td>
<td></td>
</tr>
<tr>
<td>W Dec 7</td>
<td>Th Dec 8</td>
<td>W(CNAr’Ar₆)SCXRD, single-crystal solid-state molecular structure determination of a student’s crystalline W(CNAr’Ar₆)</td>
<td>Review course F Dec 9: Simple SCXRD report</td>
<td></td>
</tr>
</tbody>
</table>

**College of Liberal Arts and Sciences (CLAS) Course Policies**

**Attendance and Absences**

Attendance at lectures and laboratory sessions is expected; in case of documented illness or University-sanctioned event, Prof. Messerle can review missed lecture to the student in drop-in office hours. Unlike other lab courses, missed laboratory sessions cannot be made up; if missed for a valid reason, Prof. Messerle will alter the nature of the lab report to cover this contingency. University regulations require that students be allowed to make up examinations which have been missed due to illness or other unavoidable circumstances. Students with mandatory religious obligations or UI authorized activities must discuss their absences with Prof. Messerle as soon as possible. Religious obligations must be communicated within the first three weeks of classes.

**Exam Policies**

**Communication: UI Email**

Students are responsible for all official correspondences sent to their UI email address (uiowa.edu) and must use this address for any communication with instructors or staff in the UI community.
Other Expectations of Student Performance

Prof. Messerle and all Teaching Assistants expect civil behavior between students and instructors and between students, with consequences for any disturbances of the class. Students have the right to a distraction-free learning environment. Students are expected to help each other learn and to contribute overall to the learning environment of the course. Arriving prepared for class is part of this expectation.

University of Iowa's Chemistry Department Educational Program Outcomes

Our Values

Our Department is committed to maintaining excellence in teaching and mentoring and achieving maximal learning benefit for each student. In order to do so, we as a Department must determine those things that make an undergraduate education successful, and then determine the things we do well all the time, some of the time, and rarely.

Program Outcomes

The graduate with a Bachelor’s Degree in Chemistry will be able to use their knowledge and skills obtained in the educational program to demonstrate:

Knowledge and Understanding of Chemistry. You should be able to:

- Master major concepts, theoretical principles, and experimental findings in chemistry, including fundamental concepts in each of the subdisciplines of Chemistry (organic, inorganic, analytical, physical, and biological)
- Understand the relationships between molecular structure and physical/chemical properties
- Understand the relationships between the microscopic, macroscopic, and symbolic descriptions of matter and the changes that matter can undergo
- Understand the conditions that affect molecular stability and factors that control the rates of molecular change

Laboratory Skills. As evidence of laboratory skills, you should be able to:

- Assess chemical and procedural hazards involved in laboratory work, including retrieval and analysis of MSDS (material safety data sheet) information
- Use proper personal protective equipment (PPE), responsibly handle and dispose of chemicals, and prepare for emergencies to minimize the risks associated with laboratory work
- Maintain a clearly-organized laboratory notebook as a record of experimental procedures and findings
- Use a variety of synthesis techniques, such as using principles of green chemistry, air-sensitive compound manipulation, solid-state and material synthesis methods, and microscale methods
- Use instrumentation and laboratory techniques to separate, purify, identify, quantify, and characterize chemical species
- Use computers as tools for data acquisition, management, and analysis

Scientific Reasoning. As evidence of an ability to reason scientifically, you should be able to:

- Pose scientific questions
- Plan and carry out scientific investigations
- Analyze data in order to make inferences about chemical and physical behaviors and properties, and construct scientific arguments to support conclusions
- Use scientific theory and/or interpretations of experimental results to explain chemical phenomena in the context of health, energy, technology, environment, agriculture, etc.
- Use mathematics and computational methods to understand and predict chemical behavior
- Identify and quantify uncertainties in measurements and limitations in methods
- Use graphs, diagrams, and other models to communicate chemical information to others
Chemical Information Skills: As evidence of these skills, you should be able to:

- Use modern library search tools such as the American Chemical Society (ACS) Chemical Abstracts Service’s SciFinder to locate and retrieve chemical literature and information including reported molecular syntheses, reactivities, characterization data, structures, physical properties, toxicities, etc.
- Read, analyze, and critically evaluate journal articles, meeting abstracts, and books in various subfields of chemistry
- Reference and cite chemical literature appropriately using ACS or other citation styles
Professional Skills: As evidence of professional skills, you should be able to:
- Report scientific findings in oral presentations in a clear and organized fashion using appropriate visual tools
- Describe experimental work and scientific findings in written reports, for instance through formal laboratory reports or technical memos
- Communicate results of scientific work to non-technical audiences
- Work collaboratively with peers to plan and conduct experiments, interpret chemical information, and solve problems
- Engage in responsible and ethical scientific conduct such as appropriately citing sources, reporting findings accurately such that others can duplicate the scientific work, and not plagiarizing the work of others

University Policies
- Accommodations for Students with Disabilities
- Basic Needs and Support for Students
- Classroom Expectations
- Exam Make-up Owing to Absence
- Free Speech and Expression
- Mental Health
- Military Service Obligations
- Non-discrimination
- Religious Holy Days
- Sexual Harassment/Misconduct and Supportive Measures
- Sharing of Class Recordings