

ADVANCED INORGANIC CHEMISTRY LABORATORY

Fall 2000

9:30AM-12:30PM - Wednesday

This course is an in-depth laboratory study of Inorganic Chemistry using modern microscale and macroscale inert atmosphere techniques. The student will study synthetic main group chemistry, coordination chemistry, and organometallic chemistry. Additionally, various spectroscopic methods will be employed for both characterization of the synthetic products and investigation of physical properties.

Instructors:

Course Instructor: Dr. Kelley J. Donaghy Office: Beeghly 310
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Laboratory Coordinator: Jolanta Coleman
Beeghly Hall Stockroom: Phone 885-1755

Required Materials:

Laboratory Notebook: A notebook with carbons (or a carbonless equivalent) and numbered pages. Carbon copies of notes is not acceptable. May be used for more than one class if other instructor agrees. These books are available from Student Affiliates and AXΣ for \$10.00.

Library Copy Card or Prepaid Departmental Card

(Departmental card: Prepay Arij \$5.00 or the equivalent of 100 copies, keep track of what you use on the form in the office, at the end of the semester if you have not used the full \$5 the extra will be returned, if you use more then pay accordingly.)

Eye protection: We will provide some but for your comfort it is suggested that you purchase your own. They must conform to OSHA regulations, for further assistance please see the lab coordinator.

Grading:

The grades for this course will be based on your final report, your technique, a mid-term exam and one final exam. The final report will be worth 80% of your grade, technique (general preparedness and independence) worth 5% and the midterm will constitute the remaining 15%.

More information about the format for this report will be forthcoming in October, but be aware that your notebook and your originality will be part of your grade. I do not want carbon copies of your labmates work and experiments. Choose your own targets based on your own interests! Your lab notebook should be written in the style of the provided example and signed by your TA at the end of each lab period. The carbons should then be put in my mailbox or handed to me, do not give me unsigned papers. Please start a new page for every day you are in lab, simply write at the top of the page that the experiment is continued from the previous date and on what page that information can be found.

Class Procedure:

As this is a graduate level laboratory, it will be completely self-directed and independent. Unlike your previous labs where your TA's have pulled together the materials necessary, you will be responsible for checking that you have the necessary chemicals and equipment for each laboratory you choose to undertake. Most of the chemicals have been organized for you and can be found in red milk-crates in room 205. Other chemicals are in the

flammable liquids cabinet in 205 and even more can be found with the help of your TA and Ms. Coleman in the stockroom. One week before you begin each lab you are required to show me your copy of a catalog entry for each main ingredient (exclude solvents), a copy of the MSDS sheets for each chemical (include solvents) you are going to use (Please don't just copy the MSDS sheets, actually read them!) **AND** an outline of your planned procedures. We will then discuss your plans, I will show you any special techniques and send you on your way with my initials on your procedure and MSDS sheets. **Anyone caught working without pre-approved procedures and MSDS sheets will be given an F for the course.** This is, of course, a safety issue! A list of reference material is included here for possible sources of standard preparations of the listed compounds.

You will be expected to make 2 compounds from the "easy" list, 2 compounds on the "hard" list, do 1 kinetic study and 1 catalytic cycle. This will mean that you will have at least a 6 part experimental section in your final report, but be aware that compounds on the "hard" list will usually involve more than one step which will mean making and characterizing more than one compound. Your compounds will be graded on their spectroscopic data, yield and purity. Therefore, for each compound you make you will be expected to obtain all pertinent spectroscopic data (IR, NMR, UV/VIS, GC/MS) and the melting point if applicable. Figuring out what is pertinent and applicable may prove to be one of your biggest challenges. If the data doesn't fit, you may want to do the reaction again. Within the first few weeks I would like to have all of you checked out on all of the above instrumentation, please let me know what instruments for which you are a "certified" user. The Labworks workstation may also be useful for some work.

Synthetic Targets (please do not plan on using more than 250mg of any limiting reagents)

Easy

Acetylferrocene
Metal Acetylacetonates
Silly Putty
Cis-Platin
(CO)₂(η-MeC₅H₅)PPH₃Mn

Hard

Ferrocene
Nickel Dicarbollide
Allyl-palladium
Liquid Crystals
1,10-phenanthroline-5,6-quinone via cobalt complex

Kinetics Experiment

Fluxionality of Allylpalladium
Cr(III) kinetics

Catalytic Cycle

Palladium Cross-Coupling
Wilkinson's Catalyst

Suggested Bibliography:

For Inorganic Background Reading

Advanced Inorganic Chemistry: F. Albert Cotton [and] G. Wilkinson. American University Library.
Call#: QD151.C64

Bioinorganic Chemistry : an introduction, Ei-ichiro Ochiai. WRLC center Call#: QP514.2.O25

Oxford Chemistry Primers – “Inorganic Materials Chemistry”, “d-Block Chemistry”, “Inorganic Chemistry in Biology”, “Organometallics I and II” - authors vary they are published by Oxford University Press (These small books usually cost about \$12 directly from Oxford and will be useful in the lecture part of this course next fall, in fact if I am the lecturer they will be the textbook!).
<http://www.oxford.com>

For Inorganic Laboratory Techniques and Procedures:

Microscale Inorganic Chemistry : a comprehensive laboratory experience, Zvi Szafran, Ronald M. Pike, Mono M. Singh. George Mason's Library. Call#: QD155.S96

Synthesis and Technique in Inorganic Chemistry : a laboratory manual, Robert J. Angelici and Gregory S. Girolami. George Mason's Library Call #: QD155.G57

Microscale and Macroscale Organic Chemistry: a laboratory manual, Williamson (required for Organic Chemistry)

Primary Literature References for Up-to-Date Examples and Background:

Journal of Chemical Education, Stacks in Chemistry Building and AU Library. On-line search engine: <http://jchemed.chem.wisc.edu/Journal/Search/index.html>

Journals: Inorganic Chemistry, Organometallic Chemistry, Chemistry of Materials, Journal of Organometallic Chemistry, Journal of the Chemical Society: Dalton Transactions, Journal of the American Chemical Society.

We can also arrange to have Martin Shapiro (the science reference librarian) talk to us about searching Chemical Abstracts on-line in the library. Please let me know if this is something you would like me to set up.

Very Tentative Laboratory Schedule

31-Aug	Introduction, Check-in, Safety and Coordination Chemistry Lecture
2-Sep	Organometallic Chemistry and Materials Lecture
6-Sep	A little more lecture if necessary!
13-Sep	Begin Labs provided you have valid Procedures, Catalog pages and MSDS sheets
11-Oct	Midterm Exam
29-Oct	No Classes, Thanksgiving Day
5-Dec	Final Report Due

Academic Integrity:

All research and laboratory work should be your own. Collaboration beyond "helpful suggestions" is a violation of the Academic Conduct Code. "Helpful suggestions" are suggestions to guide your colleague not to tell them what to do or flat out telling them what you did. Standards of academic conduct are set forth in the University's Academic Integrity Code, which can be found in the manual of Academic Regulations on page 99 or obtained from Office of University Registrar. By registering, you have acknowledged your awareness of the Academic Integrity Code and you are obliged to become familiar with your rights and responsibilities as defined by the Code. Violations of the Academic Integrity Code will not be treated lightly, and disciplinary actions will be taken should such violations occur. This includes extremely serious academic offense. Allegations of cheating will be referred to the Undergraduate Dean of the College of Arts and Science for appropriate action. Please see me if you have any questions about the academic violations described in the Code or as they relate to particular requirements of this course.