

Inorganic Chemistry Laboratory
CHE 320L
Northern Kentucky University

Spring 2006
R 1:40 - 5:40 P.M. and other times
SC461

Prerequisite: Organic Chemistry, CHE 310 and Organic Chemistry Lab, CHE 310L

Corequisite: Inorganic Chemistry, CHE 320

Instructor: J. H. Niewahner, NS205
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Office Hours: MWR 10:00 – 11:00 AM; T 2:00 – 3:00 PM; Others by appointment

Faculty Website: <http://www.nku.edu/~niewahnerj/>

Web Access: This course requires students to use and access various internet resources such as email and Blackboard.

Course Objectives

The objectives of this course are for the student to learn a) techniques used in the synthesis and isolation of air-stable and air-sensitive coordination and organometallic compounds using open-bench, airless glassware, and glovebox techniques, b) to use thin-layer and column chromatography, IR, NMR, magnetic susceptibility, absorption and emission spectroscopy, and polarimetry in identifying and characterizing coordination and organometallic compounds, and c) to keep proper laboratory records and write reports using the style approved by the American Chemical Society. Most experiments will be carried out on the microscale.

Required Text

Szafran, Zvi; Pike, Ronald M.; Singh, Mono M.; *Microscale Inorganic Chemistry*, John Wiley and Sons, Inc: New York, 1991

Additionally Recommended Text

Mohrig, J.R.; Hammond, C.N.; Schatz, P.F.; Morrill, T.C.; *Techniques in Organic Chemistry*, W.H. Freeman and Company: New York, 2003 (This book was required in the Organic Chemistry course).

Other Required Material

Safety glasses, paper towels, examination gloves, Record Book, Hayden-McNeil Publishers, Inc.

Grading

Students grades will be based on their results, reports, and lab record book.

Locker and Equipment Maintenance

You are required to keep all your equipment cleaned and in good order. Make sure all your locker equipment is returned to your locker at the end of the period. You will be penalized for each piece of missing equipment or glassware.

Lab Record Book

A record of all procedures, data and observations, calculations, and conclusions is to be kept in the Lab Record Book. The format is described in the lab manual by Szafran, et.al., p. 31-34, and in the manual by Mohrig, et.al., p 24-25. Students must record what they actually did rather than copy procedures written

in the lab manual. Also, it is important to record original data rather than mental calculations made from the data. At the end of each day of laboratory work, copies of all data recorded are to be handed in to the instructor.

Reports

Experiments will require either a Formal Report or a Short Report. Formal Reports are to be done for experiments 24B, Preparation of $[\text{Cu}(\text{Me}_2\text{im})_2]\text{PF}_6$, and the Literature Preparation. Formal Reports are to use the template for Inorganic Chemistry that can be found in Course Documents section of Blackboard. When using this template you simply have to change the title, table headings, and text to fit the experiment. Short Reports will have a cover page that includes your name, the title of the experiment, and the date of the report. Following the cover page will be an abstract of the experiment, a statement of the purpose of the experiment, balanced chemical equations for all reactions, an experimental section, a results section, and conclusions. All spectra are to be attached at the end of either report. Answers to assigned questions at the end of the experiment are to be attached to either report after the spectra. Plagiarism will result in a grade of zero for that experiment. Reports are due one week after the scheduled completion of the experiment.

Samples

All compounds synthesized are to be submitted along with the report. Samples are to be identified with your name and the name of the sample. The NMR and IR are to be taken of all compounds synthesized unless instructed otherwise.

Missed Labs

Missed labs will generally be assigned a grade of zero. Only under extreme circumstances will a student be allowed to make up a lab experiment.

Grading Scale

Experiments	80 %
Exam	20 %
<u>Percentage</u>	<u>Letter Grade</u>
90 - 100	A
80 - 89	B
70 - 79	C
60 - 69	D
0 - 59	F

Policies of the Department of Chemistry at Northern Kentucky University

- All items on syllabi are subject to change by the instructor.
- Students are responsible for reading and understanding all items on the syllabi. Any items not understood must be brought to the attention of the instructor within the first two weeks of class.
- The work you will do in any course is subject to the Student Honor Code. The Honor Code is a commitment to the highest degree of ethical integrity in academic conduct, a commitment that, individually and collectively, the students of Northern Kentucky University will not lie, cheat, or plagiarize to gain an academic advantage over fellow students or avoid academic requirements.

- Cheating will not be tolerated. In accordance with the Code of Student Rights and Responsibilities, faculty members have the right to determine actions to be taken when a student is caught cheating.
- Faculty members reserve the right to dismiss or to have removed a disruptive student from their classrooms.

Other

The Honor Code can be accessed at

http://www.nku.edu/~deanstudents/student_rights/honor_code.htm. Students caught cheating or plagiarizing will receive a grade of zero for that test or assignment and may be given an F for the semester.

Students are asked to turn off their cell phones during class. If you expect an emergency call please notify your instructor. Under no circumstance will students be allowed to use their cell phones during a test. Using a cell phone during a test will be considered cheating.

Students with disabilities who require accommodations (academic adjustments, auxiliary aids or services) for this course must register with the Office of Disability Services. Please contact the disability service office in University Center Suite 320 or by calling (859) 572-6373 for more information. Verification of your disability is required in the disability services office for you to receive reasonable academic accommodations. Visit the disability services website at www.nku.edu/~disability.

Tentative Schedule of Experiments Spring 2006

<u>Week</u>	<u>Experiment</u>	<u>Experiment Title (Point value of experiment)</u>
Jan. 12	Check in.	Intro to Techniques (Chapters 5 and 6); IR and NMR
Jan. 19	Exp. 22A	Preparation of Tris(2,4-pentanedionato)chromium(III) (100) (Effect of delocalization on carbonyl absorption frequency)
Jan. 26	Exp. 24B.	Synthesis of Copper(II) Acetate Monohydrate. (Formal Report) (150) (IR and Magnetic Susceptibility)
Feb. 2	Handout	Kinetics of Aquation of $[\text{Co}(\text{NH}_3)_5\text{Cl}]^+$ (100)
Feb. 9	Exp. 39	Synthesis of Allylpalladium chloride: Molecular Fluxionality (150)
Feb. 16	Exp. 39	Synthesis of Allylpalladium chloride: Molecular Fluxionality
Feb. 23	Handout	Introduction to Vacuum Line Techniques
Mar. 2	<i>JACS</i> , 1993 , 115, 11259	Synthesis of $[\text{Cu}(\text{Me}_2\text{im})_2]\text{PF}_6$. (Formal Report) (Glovebox)
Mar. 9	Spring Break	No Lab
Mar. 16	<i>JACS</i> , 1993 , 115, 11259	Synthesis of $[\text{Cu}(\text{Me}_2\text{im})_2]\text{PF}_6$. (Formal Report) (150) (Glovebox)
Mar. 23	Handout	Resolution of Tris(ethylenediamine)cobalt(III) Enantiomers (150) (Separation of enantiomers. Optical activity)
Mar. 30	Handout	Resolution of Tris(ethylenediamine)cobalt(III) Enantiomers (Separation of enantiomers. Optical activity)
Apr. 6	JCE,71(1),75	Synthesis of Cu(II) Macrocyclic Complex and $\text{Mo}(\text{CO})_4(\text{H}_2\text{Mac})$ (150) Complex
Apr. 13	JCE,71(1),75	IR, ^1H -NMR, Absorption and Emission Spectra of Macrocyclic Complexes
Apr. 20	Handout	Literature Preparation [@] (Formal Report) (200) (Use of SciFinder Scholar) (Preparation of an air-sensitive compound)
Apr. 21	Handout	Literature Preparation [@] (Formal Report). Check-out
May 4		Exam 1:00 - 3:00

[@] Approved literature preparations are to be selected from among those provided by the instructor. Selections must be made by January 26. The student will carry out on-line searching in order to obtain a reference to the recent literature for the synthesis of the compound. The student will then submit a copy of the reference to the instructor along with a list of the chemicals, the amounts needed to synthesize the compound, and the amounts that need to be purchased by March 2.