

Inorganic Chemistry Laboratory
CHE 320L
Northern Kentucky University

Spring 2004
R 4:00 - 8:00 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: MW 1:00 - 1:50 A.M., TR 10:00 - 10:50 A.M.

Course Content

This course will introduce the student to many advanced laboratory techniques used in synthesizing, isolating and characterizing inorganic compounds. Students will use thin layer and column chromatographic techniques; ultraviolet-visible, infrared, and nuclear magnetic resonance spectroscopic techniques; and inert atmosphere techniques (Schlenk glassware and glovebox). Basic techniques of glassblowing (bend, straight and tee joints) will also be carried out. Most of the experiments will be carried out on the microscale.

Mailserv

All students are required to subscribe to the che320jn mailserv by the end of the first week of the semester. The Mailserv provides a way to communicate between faculty and students. Students will be responsible for any information distributed to the class by this method. Students may send questions about the class material via the Mailserv.

a) To subscribe, send an email to MAILSERV@NKU.EDU (there is no E at the end of MAILSERV). In the body of the message portion type SUBSCRIBE che320jn (one space between SUBSCRIBE and CHE...)

b) To send a message, send to che320jn@nku.edu. All subscribers will receive the message by way of email to their email address.

Required Text

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

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, p. 31-34. 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 at <http://paragon.acs.org/paragon/index.jsp>. 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, the name of the sample, the amount of the sample, and the date of synthesis, and the page numbers of the research notebook where information about the compound can be found. 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	1100	points
Glass Blowing	150	points
Exam	200	points

<u>Overall Average</u>	<u>Letter Grade</u>
90 – 100 %	A
80 – 89 %	B
70 – 79 %	C
60 – 69 %	D
0 – 59 %	F

Tentative Schedule of Experiments Spring 2004

<u>Week</u>	<u>Experiment</u>	<u>Experiment Title (Point value of experiment)</u>
Jan. 15	Check in.	Intro to Glassblowing
Jan. 22	Exp. 22A	Preparation of Tris(2,4-pentanedionato)chromium(III) (100) (Effect of delocalization on carbonyl absorption frequency)
Jan. 29	Exp. 24B.	Synthesis of Copper(II) Acetate Monohydrate. (Formal Report) (100) (IR and Magnetic Susceptibility)
Feb. 5	Handout	Kinetics of Aquation of $[\text{Co}(\text{NH}_3)_5\text{Cl}]^+$ (100)
Feb. 12		Glassblowing
Feb. 19	Exp. 18 Nitrate	Positive Oxidation States of Iodine: preparation of Dipyridineiodine(I) (100)
Feb. 26	Exp. 45B (100)	Variable Temperature ^{13}C -NMR Investigation of $[(\eta^5\text{-C}_5\text{H}_5)\text{Fe}(\text{CO})_2]_2$
Mar. 4	<i>JACS</i> , 1993 , 115, 11259	Synthesis of $[\text{Cu}(\text{Me}_2\text{im})_2]\text{PF}_6$. (Formal Report) (100) (Glovebox)
Mar. 11	Spring Break	
Mar. 18	<i>JACS</i> , 1993 , 115, 11259	Synthesis of $[\text{Cu}(\text{Me}_2\text{im})_2]\text{PF}_6$. (Formal Report) (Glovebox)
Mar. 25	Handout	Resolution of Tris(ethylenediamine)cobalt(III) Enantiomers (150) (Separation of enantiomers. Optical activity)
Apr. 1		Resolution of Tris(ethylenediamine)cobalt(III) Enantiomers (Separation of enantiomers. Optical activity)
Apr. 8	JCE,71(1),75	Synthesis of Cu(II) Macrocyclic Complex and $\text{Mo}(\text{CO})_4(\text{H}_2\text{Mac})$ (150) Complex
Apr. 15	JCE,71(1),75	IR, ^1H -NMR, Absorption and Emission Spectra of Macrocyclic Complexes
Apr. 22	Handout	Literature Preparation [@] (Formal Report) (200) (Use of STN on-line searching) (Preparation of an air-sensitive compound)
Apr. 29	Handout	Literature Preparation [@] (Formal Report)
May 6		Exam

[@] Approved literature preparations are to be selected from among those provided by the instructor. Selections must be made by January 30. 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 4.

Changes in Syllabus

This syllabus is subject to change. If there is any part of this syllabus that you do not understand, you must bring it to the attention of the instructor within one week of receiving this syllabus.

Student Honor Code

Students are obligated to follow the Student Honor Code. The Honor Code can be accessed at <http://www.nku.edu/~deanstudents/HonorCode.htm>