INORGANIC CHEMISTRY LABORATORY

Preparation and characterization of inorganic and organometallic compounds by modern techniques.
For students majoring in chemistry or biochemistry and incoming graduate students.

Cr. 1. Prereq: CHEM 402 (or concurrent enrollment for graduating seniors)
Fall 2023: Aug-21-2023 until Dec-14-2023

Instructor:	**Dr. Irmi Schewe-Miller**	
Email:	irmim@iastate.edu	use "CHEM 401L" in subject line
Office:	1275 Gilman Hall	
Office hours:	Please make an appointment by email to meet F2F or online	

Course-Section	Time (1225 Hach Hall)	**open lab hours:**
CHEM 401L/501L-1	T 12:10 – 3:00 pm	R 12:10 – 6 pm; by appointment with a TA only
CHEM 401L/501L-2	T 3:10 – 6:00 pm	R 12:10 – 6 pm; by appointment with a TA only

Course Format: The lab meets once a week for a 3-hour lab-period during the entire semester; an ***optional open lab*** is available on Thursdays (by appointment with a TA only) to finish some lab work, and/or to take spectra.
Six assigned experiments, plus a challenge project, need to be completed in this course according to the posted schedule. Most experiments need to be performed individually. A few experiments are ongoing experiments and students need to schedule times to use the needed equipment. During the last four weeks in the lab, students work on a ***Challenge Project***, which is chosen from a list of suggested experiments[1], or proposed by the student. At the last meeting of this course, students give a presentation about their challenge project.
General Syllabus Statements for Iowa State University can be found in Canvas.

Objectives and Outcomes: This course serves as an introduction to advanced techniques of synthesis and characterization of a variety of inorganic materials. Students will use techniques and concepts from many areas of chemistry, make connections, and answer integrative questions. A challenge project provides a capstone experience for undergraduate students receiving a Bachelor of Science from Iowa State University.
Upon completing this course, students will be able to ...

- work with compounds from diverse areas in inorganic chemistry.
- retrieve information about hazards and safe handling of chemicals and equipment.
- use several advanced techniques for the synthesis, purification, and characterization of inorganic materials.
- evaluate and propose improvements to an experimental procedure based on their results.
- design and carry out a synthesis, purification and characterization of an inorganic material based on references in the chemical literature.
- analyze data from measurements and characterizations and assess the purity of their products.
- communicate their results and compare them to published results in the chemical literature.
- connect the properties of their products to fundamental aspects of chemical bonding and structures.

Textbooks and Supplies
Required items:

- **Immediate Access** of selected pages of "Synthesis and Technique in Inorganic Chemistry", (third edition, 1999, by G.S. Girolami, T.B Rauchfuss, and R.J. Angelici). You will be charged on your U-Bill for the copyright fees.
- PPE: safety goggles and your own lab coat
- A laboratory notebook with bound and ***numbered*** pages and removable duplicate sheets (you may continue used notebooks from other labs if they meet these requirements).

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[1] Students may also suggest an experiment for the challenge project that is not on the list, if the needed chemicals and equipment can be provided. Students need to contact the instructor well ahead of the proposal due date if they want to use this option.

Recommended items:

- "Synthesis and Technique in Inorganic Chemistry", third edition, 1999, by G.S. Girolami, T.B Rauchfuss, and R.J. Angelici; this book is also on course reserve at Parks Library. The entire book has much useful information and chapters on other experiments. Only selected pages of this book are required and accessible on Canvas. If you ***choose to buy the book***, you can "Opt–out" of the copyright fees for the select pages within the first 10 days of class and receive a refund to your U-bill.
- Any textbook of Inorganic Chemistry. I recommend the book used in Chem 301 and/or Chem 402
- The Organic Chem-Lab Survival Manual: A Student Guide to Techniques (6^{th} through 10^{th} edition acceptable) by James W. Zubrick, Wiley; (2010) ISBN-13: 978-0470494370. It has lots of useful information on laboratory techniques.
- pen, pencil, and ***your own*** permanent marker

Laboratory Notebooks: You will be using a laboratory notebook ***with bound and numbered pages and removable duplicate sheets*** for ***handwriting*** your pre-lab assignments and ***recording*** your data and observations ***during*** your experimental work. The removable duplicate sheets of your ***pre-labs*** must be submitted in time to be graded. There will be a bin in the Chemistry Majors Lab (1225 Hach Hall) to drop off your pre-labs. TAs will add important feedback to help you work in a safe and efficient manner and return your graded pre-labs at the beginning of the lab period. Use this graded pre-lab as a reference for your work in the lab. ***You may not work in the lab without a graded pre-lab***. At the end of each day in the lab you need to scan your data/observation pages in your notebook and submit them as a pdf file for the weekly ***Data and Observations*** assignments. Make sure these are well lighted, turned upright and of sufficient resolution to be easily read for grading.

How to Succeed, Learning Activities:

To successfully complete this course, students will do the following:

- work through the Unit Modules by
  - reading assigned materials
  - watching any videos posted
  - accessing resources linked in the instructions (some of these may be available only while on campus!)
  - participating in discussion topics
  - completing all quizzes
  - handwriting and turning in pre-labs
  - submitting all assignments
- work in the laboratory by
  - coming prepared to do the work
  - adhering to all safety procedures
  - taking careful notes on actual ***amounts*** of reagents and products used/obtained
  - taking careful notes on ***observations*** during the synthesis and purification steps
  - recording all parameters of spectra taken
  - cleaning their equipment and workspace
  - asking the TAs if in doubt about anything
- communicate with the instructor and TAs by
  - using the discussion boards
  - email through Canvas
  - visiting the instructor and/or TA during office hours or by appointment

NOTE: Dr. Irmi, your course instructor, suffers from hearing loss, and even though she is wearing hearing aids, her speech recognition is impaired. Her ability to understand you is severely reduced when there is background noise and several people are talking at the same time. After you speak to Dr. Irmi in person, please, summarize any important agreements that have been made and email them to Dr. Irmi for verification. Purely oral agreements are not binding ☹.

Assignments and Grading Policies: The Canvas grade book will be used. Legibility, correct spelling, and grammar will be part of the grade of your assignments. General grading rubrics will be used, as shown with the assignments. Scores of individual assignments will be shown in percentages. Different assignment categories are weighted as given below and are visible with the listing of assignments in Canvas. The standard letter-grading scheme will be used to assign final grades; however, the instructor reserves the right to adjust the grading scheme.

There are penalties for violating proper safety, waste disposal, and laboratory practices. Repeated non-compliance can result in dismissal from the laboratory. If details, such as due dates, differ between the printed syllabus and what is posted on Canvas, ***information in Canvas takes precedence.***

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Attendance: Make-ups for missed lab periods[2] are not easy to schedule and complete. Please plan any outside commitments away from class hours. You need to adhere to the chosen or assigned schedule to ensure that the required chemicals and shared equipment are available. When experiments require more than one lab period, you still need to leave your working area clean for students in other courses using this space throughout the week. Contact your instructor and TAs as soon as possible to discuss ways to make up the missed laboratory work.

Expectations: Submit your own work. Make sure to state clearly if you use data that you did not collect yourself. Any resources used to create your submissions need to be cited in the references using ACS guidelines and styles. You are encouraged to discuss questions with your classmates, TAs, and the instructor. However, submit answers to questions in your own writing.

Assignments in Units 1 - 6:

For each unit the following assignments need to be submitted for grading:

Assignment	Form	Due	Comments
pre-lab, (PL)	handwritten in your laboratory notebook: place carbon copies into bin in 1225 Hach Hall	no later than 4:00 pm on Mondays before the day of the experiment[3]	to give TAs enough time to grade them and to provide important feedback
unit quiz, (Quiz)	online: Canvas quiz	on Mondays at 11:59 pm (midnight)	to review assigned readings and experimental procedures
data and observations, (DO)	online: a scan (*.pdf) of your in-lab notes in your laboratory notebook	at the end of the lab day, Tuesdays at 11:59 pm (midnight)	no additional notes needed but your notes must be legible!
final report, (FR)	online: documents (*.docx or *.pdf)	nine days after the experiment has been performed, Thursdays at 11:59 pm (midnight)	you may insert pictures, or clear scans of hand-drawn structural formulas or schemes
structural models	online: documents (*.docx or *.pdf)	on Thursdays at 11:59 pm (midnight)	total of 2 assignments: Solids and Co(III) complexes

Use ***Canvas*** to stay informed on due dates. Late reports will result in reduced grades.

Grading Feedback: All graded assessments will be returned with feedback within 7 days of submission. Personalized feedback will be provided for each assignment. Responses to common questions and comments about unclear content will be posted in Announcements and/or the Discussion boards of each module. If you want feedback on your submission before the due date, please, submit it early and contact the TA and/or instructor to look it over.

Missed and late assignments: It is important to ***keep up with the pace*** of this course, therefore try to submit your assignments ***before the due date***.

You may not work in the lab without a graded pre-lab (PL)! There is no grace period for pre-labs. Your data and observations (DO) must be submitted by the end of each day that you worked in the lab. A scan of your notebook page(s) is all that is needed. There is a five-day grace period for DO assignments. For Final Reports (FR) a two-week grace period allows you to submit late assignments. Each day the assignment is late, the Canvas grade book deducts a certain percentage from your score. After the grace period, no submission will be accepted, and you receive a zero grade. (Late assignments will show a zero grade ***until*** they have been graded; there is no need to panic if you see a score of zero for a late assignment after submission.)

[2] ***Stay home if you are not feeling well and/or have tested positive for Covid!***

[3] If an assignment would be due on a holiday (Labor Day) it is due on Friday before that holiday

Assignments in Unit 7: Challenge Project:

Assignment	Form	Due
a brief proposal	online, document (*.docx or *.pdf)	on Thursday of week 7, at 11:59 pm (midnight),
interview	in person	choose from a list of time slots during weeks 7 – 9
proposed work schedule	online, document (*.docx or *.pdf)	on Thursday of week 9, at 11:59 pm (midnight),
one or more **pre-labs, (PL)**	hand written on paper: place into bin in 1225 Hach Hall,	no later than 4:00 pm on Monday of week 11,
4 x data and observations, (DO10 - DO-13)	online: a scan (*.pdf) of your in-lab notes in your Laboratory Notebook,	at the end of each day in the lab, on Tuesdays at 11:59 pm (midnight),
final report, (FR)	online: document (*.docx or *.pdf),	on Thursday of week 15 at 11:59 pm (midnight),
presentation	in person	on last day the class meets, Dec. 6
	online: document (*.pptx or *.pdf),	on Thursday of week 15 at 11:59 pm (midnight),

Use ***Canvas*** to stay informed on due dates. Late reports will result in reduced grades.

Challenge Project: During the last four weeks of lab work, you will work on a ***Challenge Project***. Choose your challenge project from the list at the end of this syllabus or suggest another experiment. Choose an experiment that you find interesting and about which you want to learn more. Read the summaries posted in Canvas before choosing your project. Your Challenge Project should cover four lab periods of synthetic and analytic work.
You need to submit a ***Proposal*** (due Thursday 11:59 pm of week 6) for your challenge project. This brief proposal should contain one paragraph each, on the what, why, and how of your challenge project, as well as a list of required equipment and chemicals (to give us enough time to make sure that what you need is available). You will then meet with the instructor for a scheduled ***Interview*** (during weeks 7 – 9) to discuss your proposed experiment. After the interview, you need to submit a ***Planned-Work-Schedule*** (due Thursday 11:59 pm of week 9), outlining the work by day/week. The work schedule does not have to be as detailed as a pre-lab but should show that you have a good understanding of the scope and time requirements of the proposed work. Your lab instructor must approve your proposal and your work schedule before you start working on your Challenge Project.

For each day of work on your challenge project, a graded ***pre-lab*** (PL) is necessary; you can submit a comprehensive pre-lab before the first day you work on your challenge project. If you change your plans, you need to submit a supplemental pre-lab. You need to submit a scan of your in-lab notes as ***data and observations*** (DOs) ***each week***. You will give a ***presentation*** on your Challenge Project on the last day this course meets. This presentation and your ***final report*** (FR) need to be submitted online following your presentation. You may make some final changes to the PowerPoint and the report after discussing your experiment during your presentation.

Weighting Schema:

Assignment Category	Weight for Midterm Grades	Weight for Final Grades
Pre-Labs (PL) *the lowest score is dropped*	25%	15%
Unit Quizzes (Quiz) *the lowest score is dropped*	15%	10%
Data and Observations (DO) *the lowest score is dropped*	20%	10%
Final Reports (FR) *the lowest score is dropped*	25%	15%
Structural Models	15%	10%
Challenge Project Assignments	0%	40%
EC: Contributions to Discussions and End of Semester Survey	2%	2%

Incomplete grades: In extraordinary circumstances, you can request an "Incomplete". You must be passing the course at the time of the request. You and the course instructor will negotiate and sign an incomplete contract, which needs to be resolved within one semester.

Schedule and Overview of Experiments:

Weeks	Dates	Scheduled Experiments:
1	Aug-22	Introductions – Safety – Check-in and start of first experiment (A Pre-Lab will be given to you for this day's work)
1 – 5	Aug-22 – Sep-19	**Unit 1:** **Solid State Chemistry**: Synthesis of the 1-2-3 Superconductor $YBa_2Cu_3O_{7-x}$ and structures of solids
2	Aug-29	**Models 1:** build models of related structures of solids
3 – 4	Sep-5 – Sep-12	**Unit 2:** **Main Group Chemistry:** Synthesis and characterization of a Borane-Amine adduct; FTIR and NMR spectroscopy.
5	Sep-11	**Meissner Test** of the 1-2-3 Superconductor $YBa_2Cu_3O_{7-x}$. (completes Unit 1)
5 – 6	Sep-19 – Sep-26	**Unit 3:** **Bioinorganic Chemistry:** Standard and Microwave Synthesis, Isolation and Characterization of *meso*-Tetraphenylporphyrin and Copper(II) Tetraphenylporphyrinate
7	Sep-28	**Proposal** of Challenge Project due at midnight
7 – 8	Oct-3 – Oct-10	**Unit 4:** **Coordination Chemistry:** Synthesis, Molar Conductance, and UV/Vis spectra of Co(III) Complexes
7 – 9	Oct-2 – Oct-17	**Interviews** w/Dr. Irmi about Challenge Project
9	Oct-19	**Proposed Work Schedule** of Challenge Project due at midnight
9 – 10	Oct-17 – Oct-24	**Unit 5:** **Coordination Chemistry:** Ligand Exchange and Isomerization Kinetics of the *trans*- dichlorotetraamminecobalt(III) ion
10	Oct-24	**Unit 6:** **Organometallic Chemistry:** Preparation and Use of a Titanium Metallocene: Methods of Air-Free Transfer
11 – 14	Oct-30 – Nov-27	**Unit 7:** **Challenge Project**
15	Dec-5	**Presentation of Challenge Project**

Possible Challenge Projects:

Synthesis and Characterization of Several Solids Using Powder XRD and TGA/DC: a Single Phase 1-2-3 Superconductor $YBa_2Cu_3O_{7-x}$, and a few Alkaline Earth Titanate Perovskites.
(1) Inorganic Chemistry of Group 14 elements: Synthesis of Coordination Complexes and Organometallic Derivatives of Tin and Characterization by NMR and MS; (2) Vacuum Line Synthesis and IR spectra of the Gas Germane, GeH4.
Synthesis and Characterization of $Ph_2PCH_2CH_2PPh_2$ (dppe) using Liquid Ammonia as a Solvent; Synthesis and Characterization of the $NiCl_2$(dppe) Catalyst and of another Metal -dppe Complex.
Compounds with a Metal-Metal Quadruple Bond: Synthesis and Ligand Substitution Reactions.
Synthesis and Resolution of the Optical Isomers of tris-ethylenediaminecobalt(III) iodide.
Synthesis, Reactions, and Structural Analysis of "Piano Stool" Metal-Arene Complexes.
A cobaloxime as a model of cobalamin coenzymes.
Synthesis, Characterization, and monolayer formation of gold nanoparticles.
Another experiment as suggested by student or instructor, as long as the needed reagents and equipment can be provided.

Some experiments may require you to do some work outside the regular lab period, by appointment with a TA only.