

Inorganic Chemistry Laboratory/Inorganic Preparations

Preparation and characterization of inorganic and organometallic compounds by modern techniques.

For students majoring in chemistry or biochemistry.

Cr. 1. Prereq: CHEM 402 (or concurrent enrollment for graduating seniors)

Fall 2022: Aug-22-2022 until Dec-16-2022

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

As Covid-19 variants still spread, we need to be careful in minimizing the risk of contracting and spreading the virus, inside AND outside the classroom. We encourage the following measures for your own and everyone's safety in the laboratory.

- Stay informed about the university's Covid-19 Response <https://web.iastate.edu/safety/updates/covid19>
- Get vaccinated and boosted <https://health.iastate.edu/services/covid-19-mobile-testing/>
- More information at: <https://www.cyclonehealth.iastate.edu/wellbeing-resources/> and <https://health.iastate.edu/public-health/>. All public health questions should be directed to publichealthteam@iastate.edu.
- In addition to laboratory PPE, such as goggles and your own lab coat, you are **encouraged** to wear a **facemask**.
- Whenever possible please maintain a 6-foot distance to others in the lab.
- Avoid assembling near instruments, balances, or hoods where chemicals are dispensed, or waste is collected.
- Come prepared to the laboratory so you do not need to ask any unnecessary questions.
- However, do ask for help, when unsure about a procedure.
- Maintain a clean and sanitary environment.
- **Stay home and inform your instructor** if you do not feel well, have tested positive for Covid, or have been told to self-quarantine.*
- Be patient with the TAs and yourself.

* Please, work with your course instructor to work out a satisfactory strategy of how to complete the course in case you have to miss the lab due to illness or self-isolation measures.

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 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. At the last meeting of this course, students give a presentation about their challenge project.

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 all 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:

- ☞ Digital 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 a used notebook from other labs as long as they meet these requirements).

Recommended items:

- ☞ Face mask
- ☞ "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 in 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 of your choice. I recommend the book used in Chem 301 and 402
- ☞ The Organic Chem-Lab Survival Manual: A Student Guide to Techniques (6th through 10th edition acceptable) by James W. Zubrick, Wiley; (2010) ISBN-13: 978-0470494370
- ☞ 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.** Scans of your data/observation pages from your notebook need to be submitted as pdf files 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 there
 - 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 of **amounts** of reagents and products
 - 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 and through Canvas
 - visiting the instructor and/or TA during office hours, or connecting with the instructor or TA using Webex or Zoom

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, several

people are talking at the same time, and if you are wearing a facemask. After you speak to Dr. Irmi in person, please, summarize any important agreements and email them to her 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. Grading rubrics will be used, as shown with the assignments. Scores of individual assignments will be shown in percent. 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 in the Canvas, **information in Canvas takes precedence.**

Attendance: Make-ups for missed lab periods* 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. ***Stay home if you are not feeling well and/or have tested positive for Covid!** 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. 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 on paper: place into bin in 1225 Hach Hall	no later than 4:00 pm on Mondays before the day of the experiment; if Monday is a holiday, the PL is due on the preceding Friday	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 preparation
data and observations, (DO)	online: a scan (*.pdf) of your in-lab notes in your Laboratory Notebook	at the end of the day in the lab, on Tuesdays at 11:59 pm (midnight)	no additional notes needed, but must be legible
final report, (FR)	online: documents (*.docx or *.pdf)	nine days after the experiment has been performed, on 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 the **calendar in 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 the due date. 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.

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

Assignments in Unit 7 Challenge Project:

Assignment	Form	Due
proposal	online, document (*.docx or *.pdf)	on Thursday Sep. 29, 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 Oct. 20, at 11:59 pm (midnight),
pre-lab, (PL)	hand written on paper: place into bin in 1225 Hach Hall,	no later than 4:00 pm, on Monday, Oct. 31,
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 Dec. 8 at 11:59 pm (midnight),
presentation	in person	on last day the class meets, Dec. 6
	online: document (*.pptx or *.pdf),	on Thursday Dec. 8 at 11:59 pm (midnight),

Use the *calendar in 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 given 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 may have to submit a supplemental Pre-lab. Each week you need to submit a scan of your in-lab notes as **data and observations** (DOs). 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) <i>the lowest score is dropped</i>	25%	15%
Unit Quizzes (Quiz) <i>the lowest score is dropped</i>	15%	10%
Data and Observations (DO) <i>the lowest score is dropped</i>	20%	10%
Final Reports (FR) <i>the lowest score is dropped</i>	25%	15%
Structural Models	15%	10%
Challenge Project Assignments	0%	40%
EC: Contributions to discussions and End of Semester Survey to help me make the course even better	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-23	Introductions – Safety – Check-in and start of first experiment (A Pre-Lab will be given to you for this day's work)
1 – 4	Aug-23 – Sep-13	Unit 1: Solid State Chemistry: Synthesis of the 1-2-3 Superconductor $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ and structures of solids
3 – 4	Sep-06 – 13	Unit 2: Main Group Chemistry: Synthesis and characterization of a Borane-Amine adduct; FTIR and NMR spectroscopy.
4	Sep-13	Meissner Test of the 1-2-3 Superconductor $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$. (completes Unit 1)
5 – 6	Sep-20 – 27	Unit 3: Bioinorganic Chemistry: Microwave Syntheses, Isolation and Characterization of <i>meso</i> -Tetraphenylporphyrin and Copper(II) Tetraphenylporphyrinate
6	Sep-29	Proposal of Challenge Project due at midnight
7 – 8	Oct-04 – 10	Unit 4: Coordination Chemistry: Synthesis, Molar Conductance, and UV/Vis spectra of Co(III) Complexes
7 – 9	Oct-04 – 20	Interviews w/Dr. Irmi about Challenge Project
9	Oct-20	Proposed Work Schedule of Challenge Project due at midnight
9 – 10	Oct-18 – 25	Unit 5: Coordination Chemistry: Ligand Exchange and Isomerization Kinetics of the <i>trans</i> - dichlorotetraamminecobalt(III) ion
10	Oct-25	Unit 6: Organometallic Chemistry: Preparation and Use of a Titanium Metallocene: Methods of Air-Free Transfer
11 – 14	Nov-01 – 29	Unit 7: Challenge Project
15	Dec-06	Presentation of Challenge Project

Possible Challenge Projects:

Synthesis of a single phase 1-2-3 Superconductor $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ and characterization by X-ray Diffraction; Synthesis and characterization of alkaline earth titanate perovskites.
Inorganic chemistry of group 14 elements: Synthesis of Coordination Complexes and Organometallic Derivatives of Tin and Characterization by NMR and MS; Vacuum line synthesis and IR spectra of germane, GeH_4 .
Synthesis and characterization of $\text{Ph}_2\text{PCH}_2\text{CH}_2\text{PPh}_2$ (dppe) using Liquid Ammonia as a Solvent; Synthesis and characterization of the $\text{NiCl}_2(\text{dppe})$ 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 need a day of more than 3 hours in the laboratory and/or require you to do some work outside the regular lab period.

General Syllabus Statements for Iowa State University:

Freedom of Speech and Academic Freedom: Iowa State University supports and upholds the First Amendment protection of [freedom of speech](#) and the principle of [academic freedom](#) in order to foster a learning environment where open inquiry and the vigorous debate of a diversity of ideas are encouraged. Students will not be penalized for the content or viewpoints of their speech as long as student expression in a class context is germane to the subject matter of the class and conveyed in an appropriate manner.

Academic Dishonesty: The class will follow Iowa State University's policy on academic misconduct ([5.1 in the Student Code of Conduct](#)). Students are responsible for adhering to university policy and the expectations in the course syllabus and on coursework and exams, and for following directions given by faculty, instructors, and Testing Center regulations related to coursework, assessments, and exams. Anyone suspected of academic misconduct will be reported to the [Office of Student Conduct in the Dean of Students Office](#). Information about academic integrity and the value of completing academic work honestly can be found in the [Iowa State University Academic Integrity Tutorial](#).

Accessibility Statement: Iowa State University is committed to advancing equity, access, and inclusion for students with disabilities. Promoting these values entails providing reasonable accommodations where barriers exist to students' full participation in higher education. Students in need of accommodations or who experience accessibility-related barriers to learning should work with Student Accessibility Services (SAS) to identify resources and support available to them. Staff at SAS collaborate with students and campus partners to coordinate accommodations and to further the academic excellence of students with disabilities. Information about SAS is available online at www.sas.dso.iastate.edu, by email at accessibility@iastate.edu, or by phone at 515-294-7220.

Discrimination and Harassment: Iowa State University does not discriminate on the basis of race, color, age, ethnicity, religion, national origin, pregnancy, sexual orientation, gender identity, genetic information, sex, marital status, disability, or status as a U.S. Veteran. Inquiries regarding non-discrimination policies may be directed to Office of Equal Opportunity, 3410 Beardshear Hall, 515 Morrill Road, Ames, Iowa 50011, Tel. 515-294-7612, Hotline 515-294-1222, email eooffice@iastate.edu

Prep Week: This class follows the Iowa State University Prep Week policy as noted in section 10.6.4 of the [Faculty Handbook](#).

Religious Accommodation: Iowa State University welcomes diversity of religious beliefs and practices, recognizing the contributions differing experiences and viewpoints can bring to the community. There may be times when an academic requirement conflicts with religious observances and practices. If that happens, students may request reasonable accommodations for religious practices. For students, you should first discuss the conflict and your requested accommodation with your professor at the earliest possible time. In all cases, you must put your request in writing. The instructor will review the situation in an effort to provide a reasonable accommodation when possible to do so without fundamentally altering a course. You or your instructor may also seek assistance from the Dean of Students Office <http://www.dso.iastate.edu/sa/> at 515-294-1020 or the Office of Equal Opportunity <http://www.eoc.iastate.edu/> at 515-294-7612.

Contact Information for Academic Issues: If you are experiencing, or have experienced, a problem with any of the above issues, email academicissues@iastate.edu