

Inorganic Chemistry Laboratory

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

Cr. 1. Prereq: CHEM 402

Spring 2021: Jan-25-2021 until Apr-30-2021

Instructor:	Dr. Irmi Schewe-Miller		Alan Medina-Gonzales medinago@ia...
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Course-Section	Time (1225 Hach Hall)	Teaching Assistants	open lab hours:
CHEM 401L-1	T 9:00 – 11:50 am	Allie*, Alan M & Arka S	R 9:00 – 11:50 am
CHEM 401L-2	T 12:10 – 3:00 pm	Allie*, Arka S & Charles W	by appointment with a TA only
CHEM 401L-4	R 12:10 – 3:00 pm	Allie*, Charles W & Alan M	

* Allie works part-time for this lab and will only attend the first 60 minutes of each section.

Due to the COVID-19 pandemic, you are expected to abide by the following measures for your own and everyone's safety in the laboratory.

- The laboratory is limited to less than 50% capacity.
- In addition to laboratory PPE, such as goggles and your own lab coat, you need to wear a facemask.
- You are expected to maintain a 6-foot distance to others in the lab whenever possible.
- Please follow directions for circulation through the lab where given.
- Do not assemble 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 or have been told to self-quarantine.*
- Be patient with the TAs and yourself, as we are all adjusting to the "new normal" these days.

* 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.

Students are responsible for abiding by the university's **COVID-19 health and safety expectations**
[\(dso.iastate.edu/guidance-for-supporting-community-expectations-during-covid-19-pandemic\)](https://dso.iastate.edu/guidance-for-supporting-community-expectations-during-covid-19-pandemic)

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. All experiments need to be performed individually. A few experiments are ongoing experiments due to equipment availability. During the last four weeks in the lab, you will work on a **Challenge Project**, which you will choose from a list of suggested experiments. At the last meeting of this course, you will give a presentation about your 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 of various areas in inorganic chemistry.
- ☞ retrieve information about hazards and safe handling of chemicals and equipment.
- ☞ use a number of 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:

- ☞ Face mask
- ☞ Select pages of "Synthesis and Technique in Inorganic Chemistry", (third edition, 1999, by G.S. Girolami, T.B Rauchfuss, and R.J. Angelici) on RedShelf. 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.
(NOTE: The Electronic Lab Notebook (ELN) "LabArchives™" is not used in this course.)

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 select pages of this book are required and accessible in RedShelf through 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: "Inorganic Chemistry" by Weller, Overton, Rourke, and Armstrong; 7th ed. 2018.
- ☞ 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 a 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. NOTE: you are asked to **handwrite** the pre-labs, so you do not just "copy and paste" important information. By handwriting the pre-lab, you will be better prepared to work in the lab. 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
 - hand-writing 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 work space
 - 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 Webex office hours
 - connecting with the instructor or TA using Webex or Zoom

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 based on outcomes 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*. 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 is 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!*** 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 and information used to create your submissions need to be cited in the references. You are encouraged to discuss questions with your classmates, TAs, and instructor (Discussion Boards). 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 lab notebook; place duplicate pages into the bin in 1225 Hach Hall	no later than 4:00 pm on Mondays/Wednesdays before the day of the experiment	to give TAs enough time to grade them and to provide important feedback
unit quiz, (Quiz)	online: Canvas quiz	on Mondays/Wednesdays 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/Thursdays 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/Saturdays 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/Saturdays 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 two-day grace period for DO assignments. For Final Reports (FR) a two-week grace period allows you to submit late assignments. The grade book deducts a percentage from your score for each day the assignment is late. 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 by the TA; 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:

The following assignments need to be submitted for grading:

Assignment	Form	Due
proposal	online, document (*.docx or *.pdf)	on Thursday/Saturday of week 6: Mar-4,6, at 11:59 pm (midnight),
proposed work schedule	online, document (*.docx or *.pdf)	on Thursday/Saturday of week 8: Mar-18,20, 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/Wednesday of week 10: Mar-30, Apr-1
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 the day in the lab, on Tuesdays/Thursdays at 11:59 pm (midnight),
final report, (FR)	online: document (*.docx or *.pdf),	on Thursday/Saturday Apr-29, May-1, at 11:59 pm (midnight)
presentation	in person	on last day the class meets
	online: document (*.pptx or *.pdf),	on Thursday/Saturday Apr-29, May-1, 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 know more about. 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 a paragraph each, on the what, why, and how of your challenge project. You will then meet with the instructor for a scheduled **Interview** (during weeks 6-8) to discuss your proposal. After the interview, you need to submit a **Planned-Work-Schedule** (due Thursday/Saturday at 11:59 pm of week 8), 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 working on your challenge project. Each week you need to submit a scan of your in-lab notes as **data and observations** (DOs). On the last day, this course meets you will give a **presentation** on your Challenge Project. This presentation and your **final report** (FR) need to be submitted online following your presentation.

Weighting Schema

Assignment Category	Weight
Pre-Labs (PL) <i>the lowest score is dropped</i>	15%
Unit Quizzes (Quiz) <i>the lowest score is dropped</i>	10%
Data and Observations (DO) <i>the lowest score is dropped</i>	10%
Final Reports (FR) <i>the lowest score is dropped</i>	15%
Structural Models	10%
Challenge Project Assignments	40%
Total	100%
EC: End of Semester Survey to help me make the course even better	1%

Incomplete grades: In extraordinary circumstances, you can request an "Incomplete". You must be at a passing grade of 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	Jan-26, 28	Introductions - Safety - Check-in and start of first experiment
1 - 5	Jan-26 - Feb-25	Unit 1: Solid State Chemistry: Synthesis of the 1-2-3 Superconductor $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$.
2	Feb-2, 4	Unit 2: Main Group Chemistry: Synthesis and IR spectroscopic characterization of a Borane-Amine adduct
3 - 4	Feb-9 - Feb-18	Unit 3: Bioinorganic Chemistry: Microwave Synthesis of <i>meso</i> -Tetraphenylporphyrin and Copper(II) Tetraphenylporphyrinate
5	Feb-23, 25	Meissner Test of the 1-2-3 Superconductor $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$. (completes Unit 1)
5 - 6	Feb-23 - Mar-4	Unit 4: Coordination Chemistry: Synthesis, Molar Conductance, and UV/Vis spectra of Co(III) Complexes
6	Mar-4, 6	Proposal of Challenge Project due at midnight
6 - 8	Mar-6 - Mar-20	Interviews w/Dr. Irmi about Challenge Project
7 - 9	Mar-9 - Mar-25	Unit 5: Coordination Chemistry: Ligand Exchange Kinetics of the <i>trans</i> -to- <i>cis</i> Isomerization and Aquation of the dichlorotetraamminecobalt(III) ion
8	Mar-18, 20	Proposed Work Schedule of Challenge Project due at midnight
9	Mar-23, 25	Unit 6: Organometallic Chemistry: Preparation and Use of a Titanium Metallocene: Methods of Air-Free Transfer
10 - 13	Mar-30 - Ap-22	Unit 7: Challenge Project
14	Apr-27, 29	Presentation of Challenge Project

Possible Challenge Projects:

Characterization of the 1-2-3 Superconductor $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ and of a layered Vanadyl Phosphate Compound by X-ray Diffraction; Iodometric Titration of Cu in $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$.*
Coordination Complexes and Organometallic Derivatives of Tin: Synthesis and Characterization by NMR and MS.
Preparation of $\text{Ph}_2\text{PCH}_2\text{CH}_2\text{PPh}_2$ (dppe) using Liquid Ammonia as a Solvent, and preparation of a metal complex using dppe as a ligand.
Compounds with a Metal-Metal Quadruple Bond: Synthesis and Ligand Substitution Reactions *
Synthesis and Resolution of the Optical Isomers of tris-ethylenediaminecobalt(III) chloride.
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.

* These 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.

Accessibility Statement: Iowa State University is committed to assuring that all educational activities are free from discrimination and harassment based on disability status. Students requesting accommodations for a documented disability are required to work directly with staff in Student Accessibility Services (SAS) to establish eligibility and learn about related processes before accommodations will be identified. After eligibility is established, SAS staff will create and issue a Notification Letter for each course listing approved reasonable accommodations. This document will be made available to the student and instructor either electronically or in hard-copy every semester. Students and instructors are encouraged to review contents of the Notification Letters as early in the semester as possible to identify a specific, timely plan to deliver/receive the indicated accommodations. Reasonable accommodations are not retroactive in nature and are not intended to be an unfair advantage. Additional information or assistance is available online at www.sas.dso.iastate.edu, by contacting SAS staff by email at accessibility@iastate.edu, or by calling 515-294-7220. Student Accessibility Services is a unit in the Dean of Students Office located at 1076 Student Services Building.

Academic Misconduct: The class will follow Iowa State University's policy on academic dishonesty. Anyone suspected of academic dishonesty will be reported to the Dean of Students Office (<http://www.dso.iastate.edu/ja/academic/misconduct.html>). Academic Misconduct in any form is in violation of Iowa State University Student Disciplinary Regulations and will not be tolerated. This includes but is not limited to: copying or sharing answers on tests or assignments, plagiarism, submitting a lab report for an experiment not performed, and having someone else do your academic work. Depending on the act, a student could receive an F grade on the test/assignment, F grade for the course, and could be suspended or expelled from the University. See the Conduct Code at www.dso.iastate.edu/ for more details and a full explanation of the Academic Misconduct policies.

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

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.

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.

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