# CHEM 3121—Inorganic Chemistry Lab Syllabus

UT Tyler
Spring 2024

Section 001 F 1:30-5:30 pm
Section 002 Th 5:30-9:30 pm
RBS 2015/4012

## About This Course:

Students will learn a variety of practical techniques in the synthesis, characterization, and handling of a variety of inorganic and organometallic compounds. Students will also learn about writing technical papers or reports of publishable quality.

*"I consider nature a vast chemical laboratory in which all kinds of composition and decompositions are formed."*

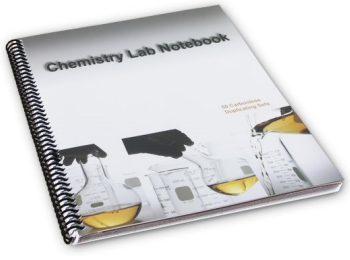
Antoine Lavoisier

## Student Learning Outcomes

- Handle laboratory glassware, equipment, and chemical reagents safely using general guidelines and basic knowledge about common hazards often encountered in a synthetic chemistry laboratory.
- Use instrumentation commonly found in a synthetic inorganic chemistry laboratory.
- Interpret laboratory results and data correctly within inherent limitations on precision and report findings in a scientific notebook using acceptable appropriate notational and descriptive content that is in turn understandable and reproducible.
- Apply procedures from literature sources to synthesize a given compound.
- Write scientific journals and reports which clearly present scientific data and which include lucid, logical conclusions based on the experimental data.

## Required Items

- Experiments will be posted on Canvas as handouts
- Lab notebook (carbonless copy or regular notebook)
- Pen (no pencils!)
- Scientific calculator
- Appropriate lab attire (INDIRECT VENT safety goggles, close-toed shoes, pants, etc.)




**Inside This Syllabus:**

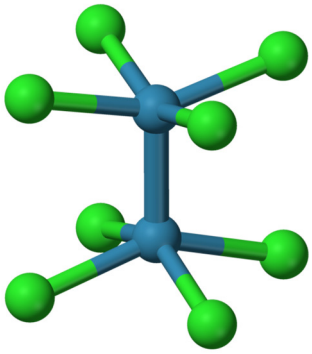


**Contact Information:**


- Dr. Jason Smee
- jsmee@uttyler.edu (best)
- 903.566.7069
- Office: RBS 3030
- Howdy (Office) Hours: MWF 9:45 –11:30 am and by appointment


# Course Requirements

- Prerequisites for this course: CHEM 1312/ 1112 (Gen Chem 2 lecture and lab).
- Co-requisite: Credit for/concurrent enrollment in CHEM 3320.
- Section 001 (Friday) meets Fridays from Jan 19 through April 19 (except Spring Break) from 1:30-5:30 p.m.
- Section 002 (Thursday) meets Thursdays from Jan 18 through April 18 (except Spring Break) from 5:30-9:30 p.m.
- Class meets in RBS 2015 before going to the organic lab (RBS 4012)
- You ***may*** need to come in outside of class time to finish an experiment. These occasions will be kept to a bare minimum.
- The "Census Date" is Monday, Jan 29th. See the University Policies in Canvas for more information regarding dropping class, grade replacement, etc.
- The last day to drop the course with a W is Monday, March 25. If you wish to drop the course, it is YOUR responsibility; failure to officially withdraw from the course will result in a grade of F.
- Attendance will NOT be taken. However, it will be obvious if you aren't there. Please notify me at your earliest convenience if you will be absent.



The octachlorodimolybdate(II) tetraanion that possesses a metal-metal quadruple bond. You will make a similar compound in lab this semester.

*"A scientist in his laboratory is not a mere technician: he is also a child confronting natural phenomena that impress him as though they were fairy tales."*

Marie Curie

# Grading

The grading scale will tentatively be based on the 90/80/70/60 (A/B/C/D) scale. You will be evaluated based on your report summaries, one full lab report, and 2 assignments. There are no exams. The weighting of the grades is as follows:

| | |
|---|---|
| Notebook and result summaries | 65% (5% for 1st summary; 15% for the others) |
| Full lab report | 20% |
| 2 outside assignments (7.5% each) | 15% |
| Total | 100% |

# Laboratory Notebook (see pp 10-11 in lab manual)

- Before lab you should have a pre-lab written up. It should have the following sections
  - Title/Date
  - Purpose
  - Overall reaction
  - Safety; this should include the name, formula, CAS #, the molar mass, density (if a pure liquid is involved), and safety hazards (irritant, corrosive, etc.)
  - Procedure:: Include all details related to conducting the experiment. Notebooks are to be written so that someone months or years later can reproduce your results. To help with calculations later you should have molar masses (and where applicable, densities of liquids) for all reagents and products (this is not necessary for solvents or rinsing/cleaning agents).
  - Reference for the procedure
- During lab you should add the following sections
  - Data/Results/Observations: Record all measurements and observations made during the lab. Be sure to note any deviations from the procedure you were given. Label data and observations clearly to avoid confusion.
  - Calculations; each experiment will include the calculations required to be in your notebook for that experiment
- Use a blue or black ink pen to record your data! No pencils! If you need to make a correction to something, especially data, draw a line through the mistake.
- At the end of lab, sign your data/observations and either I or the TA will do the same.
- Photograph your notebook pages and insert them, in order, at the end of your report.


Magnetic susceptibility balance similar to the one you will use in lab to determine the number of unpaired electrons in a transition metal complex you will synthesize.

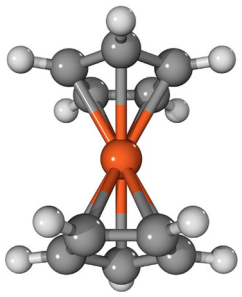
## Summary Reports

- For all experiments, except the Acylation of Ferrocene experiment, you will write up a summary of your experimental and results sections as if there were to be published in a journal.
- A sample journal article is posted on Canvas for ideas on style/presentation.
- Each summary should include the sections below
    - **Cover Page:** Title/Date/Your Name & Your Partner's Name
    - **Procedure**: Cite the procedure from the lab manual or handout. You should also write out the entire synthesis and be sure to include any deviations or modifications to the procedure. Your procedure should also identify any instrumentation used (make and model), and how samples were prepared, and the conditions under which the samples were run.
    - **Results**: Separate your results, if applicable, into the following:
        - Synthesis: describe the reaction briefly (color changes, precipitates, yields, etc.). Indicate any problems or interesting aspects of the synthesis.
        - Characterization: describe any type of spectroscopic results (IR, UV-vis, NMR, magnetic susceptibility, etc.); figures are nice. If possible you should try and assign features in your characterization (e.g. "the peak at 1.97 ppm in the $^{1}$H NMR is due to the methyl group of some residual acetic acid in the sample). For comparing multiple compounds, use tables!
    - **Discussion/Analysis**: show pertinent analytical data (i.e. titration curve) in table and/or graphical format. Indicate any equations you used in calculating an answer. If applicable compare with the known literature values (cite your references!) and note any differences. If these are significant then discuss why your results differ.
    - **References**: in the main body of the summary use superscripted numbers for the citations. Refer to this website for information on how to format your bibliography. You can also consult *The ACS Style Guide: A Manual for Authors and Editors* 3/E (call number QD8.5 .A25 2006), in the library or borrow one from a faculty member.
    - Submit your ***OWN*** summary through UniCheck on Canvas.

## Full Lab Report (Acylation of Ferrocene Experiment)

- Your report should include the following sections:
    - **Cover Page:** same as above plus an abstract (2 or 3 sentences that briefly describe your results)
    - **Introduction**: Briefly discuss the principle(s) of the experiment and give pertinent chemical equations.
    - **Procedure:** same as above
    - **Results:** same as above
    - **Discussion/Analysis:** same as above
    - **Conclusions:** any general comments regarding your results
    - **References:** same as above
- Submit your ***OWN*** report through TurnItIn on Canvas.

***Rubrics for Each Summary Report and the Full Lab Report will be provided on Canvas.***

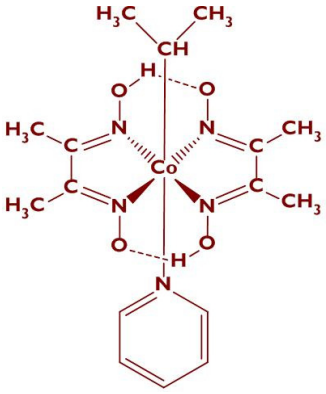


Ferrocene, $[Fe(C_5H_5)_2]$, put organometallics research into high gear. We will be attaching an acetyl group to one of the rings in the last experiment. By using cyclic voltammetry, we will see how the electronic properties of the metal changed by adding the acetyl group.

*"An investigator starts research in a new field with faith, a foggy idea, and a few wild experiments. Eventually the interplay of negative and positive results guides the work. By the time the research is completed, he or she knows how it should have been started and conducted."*

Donald Cram

The Attenuated Total Reflectance (ATR) Infrared (IR) Spectrometer you will use to analyze some of the compounds made in this lab.



A cobaloxime compound, used as a model for vitamin $B_{12}$, that you will synthesize and characterize by $^1H$ NMR.

*"Don't despise empiric truth. Lots of things work in practice for which the laboratory has never found proof."*

Martin H. Fischer

Double manifold (aka Schlenk line) often used in conjunction with an inert atmosphere glovebox to handle air-sensitive compounds.

## Summary Report Checklist

- ☐ Cover page
- ☐ Introduction (Full Lab Report only)
- ☐ Procedure (Syntheses and Physical Methods)
- ☐ Results/Analysis (plus Discussion for Full Lab Report)
- ☐ Conclusion (Full Lab Report only)
- ☐ References (superscripted numbers for citations and proper bibliographic formatting)
- ☐ Pictures of notebook pages, pasted in order, are included at the end of your report
- ☐ Electronic copy submitted through UniCheck
- ☐ Hard copy of report (optional)

## List of Experiments

- Synthesis of Copper Complexes: $K_2[Cu(ox)_2] \cdot 0.5H_2O$ (basic synthesis techniques)
- Synthesis of $[Cr_2(OAc)_4]$ (characterization by IR spectroscopy)
- $\Delta_o$ of Chromium(III) Complexes (synthesis of $[Cr(acac)_3]$, $[Cr(pic)_3]$, and $[Cr(en)_3]Cl_3$ (microwave synthesis and characterization by UV-vis spectrophotometry)
- Magnetic Susceptibility of Two Nickel Complexes (synthesis and characterization by magnetic susceptibility)
- Synthesis of $[Co(Hdmg)_2(py)(^iPr)]$ (characterization by $^1H$ NMR spectroscopy)
- Acylation of Ferrocene (separation by column chromatography, and characterization by cyclic voltammetry)

## Outside Assignments

To supplement the lecture material, you will complete 2 assignments that will constitute a total of 10% of your grade. You will complete these assignments, on your own time, outside of the normally scheduled lab time.

- The first assignment will be an introduction to reading chemical literature. A paper and a set of guided questions will be provided.
- Due Friday Feb 23 at 11:59 pm
- The second assignment will be using the CCDC (Cambridge Crystallographic Data Centre) website to obtain crystallographic data. This website allows you to visualize any crystal structures with at least one C-H bond. You can also get bond angle, bond length, and a variety of other geometric information. The assignment and links to the website will be posted as the relevant material is made available in lecture.
- Due April 12 at 11:59 pm
- If you need help or guidance, please don't hesitate to ask.

## Summary of Important Dates

- January 29 (Monday): Census Date (see Census Date section in University Policies)
- March 1 (Friday): Final Deadline to Apply for Spring Graduation
- March 11 – 15 (M – F): Spring Break, no classes
- March 25 (Monday): Last Day to Drop with a "W"
- April 18/19 (Thursday/Friday): last lab report due
- April 25/26 (Thursday/Friday): no lab

# Tentative Lab Schedule

| Date | Experiment |
|---|---|
| Jan 18/19 | Introduction and syllabus; go over Chemical Literature Outside Assignment |
| Jan 25/26 | **Must have ≥80% on Chemical and Biological Hygiene Canvas quiz in the past year**<br>Pre-lab: reminder on formatting summary reports<br>**Experiment 1: Synthesis of $K_2[Cu(ox)_2]\cdot 0.5H_2O$** |
| Feb 1/2 | **Experiment 1 summary report due**<br>Pre-lab: experiment summary, changes to procedure, brief intro to IR<br>**Experiment 2. Synthesis of a Metal-Metal Quadruple-Bonded Complex: $Cr_2(OAc)_4$** |
| Feb 8/9 | If necessary, finish **Experiment 2.** |
| Feb 15/16 | **Experiment 2 summary report due**<br>Pre-lab: experiment summary, changes to procedure, brief intro to UV-vis<br>**Experiment 3. Measuring $\Delta_o$ for Four Chromium Compounds (synthesis part only)** |
| Feb 22/23 | **Chemical Literature Outside Assignment due**<br>**Finish Experiment 3.** |
| Feb 29/Mar 1 | **Experiment 3 summary report due**<br>Pre-lab: experiment summary, changes to procedure, brief intro to magnetic susceptibility<br>**Experiment 4. Magnetic Susceptibility of Two Nickel Complexes** |
| Mar 7/8 | Go over Crystallography Outside Assignment<br>If necessary, finish **Experiment 4.** |
| Mar 14/15 | **Spring Break—no labs :)** |
| Mar 21/22 | **Experiment 4 summary report due**<br>Pre-lab: experiment summary, changes to procedure, brief intro to $^1$H NMR<br>**Experiment 5. Synthesis of a Vitamin B12 Analog: $[Co(Hdmg)_2(py)(^iPr)]$** |
| Mar 28/29 | If necessary, finish **Experiment 5.** |
| Apr 4/5 | **Experiment 5 summary report due**<br>Pre-lab: experiment summary, changes to procedure, brief intro to cyclic voltammetry<br>**Experiment 6. Acylation of Ferrocene (full lab report)** |
| Apr 11/12 | **Crystallography Outside Assignment due**<br>If necessary, finish **Experiment 6.** |
| Apr 18/19 | **Experiment 6 FULL LAB REPORT due** |
| Apr 25/26 | **No Lab This Week—Start Studying for Final Exams** |