CHEM 3121—Inorganic Chemistry Lab Syllabus

UT Tyler
Spring 2023

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."

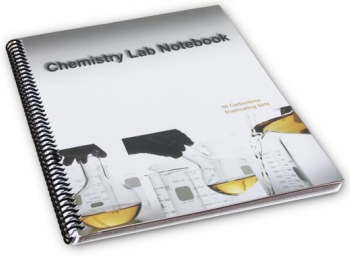
Antoine Lavoisier

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.)



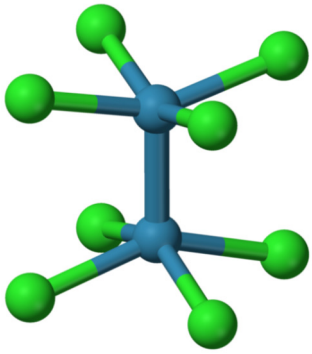
Inside This Syllabus:

Contact Information:

- Randy J. Sronce (Sections 1 and 2)
- rsronce@patriots.uttyler.edu
- 903.215.9427
- Office: RBS 2013
- Office Hours: Thursday 4-5 pm and Friday 12-1 pm or by appointment

Course Requirements

- Prerequisites for this course: CHEM 1312/ 1112 (General Chemistry 2 lecture and lab).
- Co-requisite: Credit for or concurrent enrollment in CHEM 3320.
- Class meets every week from January 9 through April 21 from 1:30-5:30 p.m. (F) and 5:30-9:30 p.m. (Th) in RBS 2015/4012, except during Spring Break.
- You may be required to come in outside of class time to finish an experiment. These occasions will be kept to a bare minimum.
- The deadline for all registrations, schedule changes, and section changes (the "Census Date") is Monday, January 23. Please see the University Policies section at the end of this syllabus for more information regarding dropping class, grade replacement, etc.
- The last day to drop the course with a W is Thursday, March 23. 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 that you are not 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

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.

Grading Scale

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:

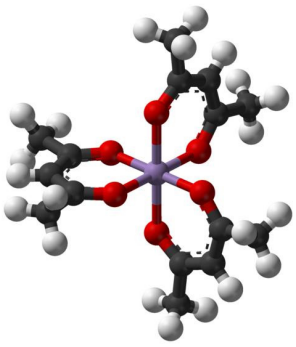
Notebook and result summaries	65% (5% for 1st summary; 15% for the others)
Full lab report	20%
2 outside assignments (7.5% each)	15%
Total	100%

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.

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.



Manganese(III) acetylacetonate, the compound you will be synthesizing and analyzing with the magnetic susceptibility balance.

"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

Full Lab Report (Acylation of Ferrocene Experiment)

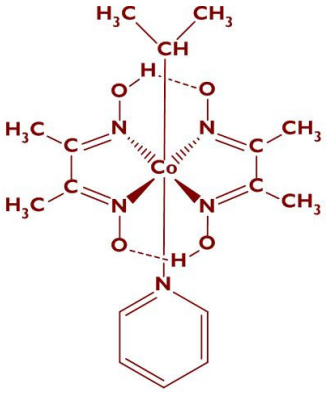
- Your report should include the following sections:
    - **Cover Page:** same as above plus an abstract (2 or 3 sentences that briefly describe your results)
    - **Introduction**: Briefly discuss the principle(s) of the experiment and give pertinent chemical equations.
    - **Procedure:** same as 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.

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

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

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)



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

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)
- Δ_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)
- Synthesis of $[Mn(acac)_3]$ (characterization by magnetic susceptibility)
- Synthesis of $[Co(Hdmg)_2(py)(^iPr)]$ (characterization by ^{1}H NMR spectroscopy)
- Acylation of Ferrocene (separation by column chromatography and characterization by cyclic voltammetry)

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

Martin H. Fischer

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. They will be due by the last day of lab (April 22 or 23, depending on your section).

- The first assignment will be an introduction to reading chemical literature. A paper and a set of guided questions will be provided.
- The second assignment will be using the CCDC (Cambridge Crystallographic Data Centre) website. This website allows you to visualize unit cells for all 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.
- If you need help or guidance, please don't hesitate to ask.

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

Summary of Important Dates

- January 23 (Monday): Census Date (see Census Date section in University Policies)
- March 1 (Wednesday): Final Deadline to Apply for Spring Graduation
- March 13 – 17 (M – F): Spring Break, no classes
- March 23 (Thursday): Last Day to Drop with a "W"
- April 20/21 (Thursday/Friday): last lab report and both outside assignments are due

Tentative Lab Schedule

Date	**Experiment**
Jan 12/13	Introduction/syllabus
Jan 19/20	Pre-lab: reminder on formatting summary reports **Experiment 1: Synthesis of $K_2[Cu(ox)_2]\cdot 0.5H_2O$**
Jan 26/27	Pre-lab: experiment summary, changes to procedure, brief intro to IR **Experiment 2. Synthesis of a Metal-Metal Quadruple-Bonded Complex: $Cr_2(OAc)_4$**
Feb 2/3	If necessary, finish **Experiment 2.**
Feb 9/10	Pre-lab: experiment summary, changes to procedure, brief intro to UV-vis **Experiment 3. Measuring Δ_o for Four Chromium Compounds (synthesis part only)**
Feb 16/17	**Finish Experiment 3.**
Feb 23/24	Pre-lab: experiment summary, changes to procedure, brief intro to magnetic susceptibility **Experiment 4. The Spin State of $Mn(acac)_3$: A Magnetic Susceptibility Study**
Mar 2/3	If necessary, finish **Experiment 4.**
Mar 10/11	Go over solid-state web assignment; due no later than Apr 21/22
Mar 16/17	**Spring Break—no labs :)**
Mar 23/24	Pre-lab: experiment summary, changes to procedure, brief intro to magnetic susceptibility **Experiment 5. Synthesis of a Vitamin B12 Analog: $[Co(Hdmg)_2(py)(^iPr)]$**
Mar 30/31	If necessary, finish **Experiment 5.**
Apr 6/7	Pre-lab: experiment summary, changes to procedure, brief intro to cyclic voltammetry **Experiment 6. Acylation of Ferrocene (full lab report)**
Apr 13/14	If necessary, finish **Experiment 6.**
Apr 20/21	**Experiment 6 FULL LAB REPORT due** **Outside assignments due**