Inorganic Chemistry Laboratory

Chemistry 390, Spring 2018

Instructor
:
Dustin Wheeler, Ph.D.
Office: HN1408
Office hours: By appointment only
Email: dustin.wheeler@hunter.cuny.edu

Course goal

Chemistry 390 is an advanced inorganic laboratory course. By the end of this course, you will have experience with the synthesis and characterization of a variety of inorganic and organometallic compounds. You will perform both micro- and macro-scale syntheses and characterize your products using analytical techniques including FTIR, NMR, UV-Visible spectroscopy, electrochemistry, and some simple methods such as melting and/or boiling points.

This course is intended to help you grow as a scientist by:
:

1. reinforcing the techniques and skills that a synthetic chemist would employ in the workplace and
2. guiding you towards the development of better reasoning and critical thinking skills for problem solving.

Books

Required: One carbon copy laboratory notebook (available in the Hunter College bookstore).
You do not need to buy a textbook for this class. I will provide handouts or post the relevant material on Blackboard for you. You should print out each lab and *must* complete a summary of the lab procedure in your notebook before you come to class.

Optional: Any inorganic text will be appropriate to use as a reference, but my favorite

is : *Inorganic Chemistry,* 5th ed. by G. Miessler, P. Fischer, and D. Tarr.

If you are interested in purchasing an inorganic laboratory book (also optional), many of our experiments will be modified from the standard inorganic laboratory lab manual by Szafran, Pike, and Sing: *Microscale Inorganic Chemistry – A Comprehensive Laboratory Experience,* 1991, John Wiley & Sons, New York, NY.

Grading

Your grade for this course will be based on 3 factors:
:

- Attendance and participation in each and every laboratory period (40%)
- A series of short laboratory assignments (lab reports) to be completed at home. (30%)
- The complete and accurate synthesis and full characterization of metalloporphyrin complexes. This will be written up as a full-scale (*JACS*-style) research paper. (30%)

: Lab writeups for experiments 2 through 6 are due at the start of the second class period following the completion lab (*e.g.,* the writeup for experiment 2 should be submitted before the start of class on Week 6, the writeup for experiment 3 on Week 8, etc.). Note the exception for Experiment 5 (due to Spring Break). Writeups should be clear, concise evaluations of the experiment, including any observations, experimental procedures, instrumental methods and instrumentation used, and any additional analyses you performed. The writeup for experiment 7 will be due by 5pm on May 29th (the Tuesday after Memorial Day).

Web site

As part of the course we will be using Blackboard. Instructions on how to access the course website on Blackboard can be found at: http://bb.hunter.cuny.edu. In addition to instructions for individual labs, I will also periodically post announcements and additional resources on Blackboard.

Attendance and Tardiness

Your attendance in each and every lab is mandatory. There will be no makeup labs offered for this course. If you are **more than 15 minutes late** for class you will not be permitted to perform the lab.

Lab Safety
:

- First and foremost you will be ***required*** to wear safety goggles at ***all*** times when in the laboratory. If you are caught without safety glasses on more than one occasion you will be asked to leave the lab.
- Open-toed shoes are not permitted in the laboratory.
- No food or drink is allowed in the laboratory at any time.
- Do not sniff or taste ***any*** of the chemicals you will be using.
- Toxic substances must be used only under the hood. You will be responsible for looking up and understanding the MSDS of **all** chemicals used in the laboratory.
- All cell phones, pagers, CD players, MP3 players, etc. must be turned off (or at the very least on silent) while in the laboratory.

Academic Dishonesty

Hunter College regards acts of academic dishonesty (*e.g.,* plagiarism, cheating on examinations, obtaining unfair advantage, and falsification of records and official documents) as serious offenses against the values of intellectual honesty.

The college is committed to enforcing the CUNY Policy on Academic Integrity and will pursue cases of academic dishonesty according to the Hunter College Academic Integrity Procedures.

Schedule of Laboratory Experiments

Date	Week	Experiment Title
1/31	1	Introduction, Experiment 1 Synthesis of silly putty
2/7	2	Experiment 2 *trans*-dichlorobis(ethylenediamine) cobalt(III) chloride
2/15	3	Experiment 2 *cis*-dichlorobis(ethylenediamine) cobalt(III) chloride
2/14	4	Experiment 2

Date	Week	Experiment Title
		Monosulfite analog of *trans*-dichlorobis(ethylenediamine)cobalt(III) chloride
2/28	5	Experiment 3 Chromium(II) acetate synthesis
3/7	6	Experiment 3 (Exp. 2 report due) Chromium(II) acetate characterization
3/14	7	Experiment 4 Spectrochemical series of chromium(III)
3/21	8	Experiment 4 (Exp. 3 report due) Spectrochemical series of chromium(III)
3/28	9	Experiment 5 Octachlorodirhenate synthesis
4/5		**No class -- Spring break!**
4/11		**Friday Schedule**
4/18	10	Experiment 6 (Exp. 4 report due) Synthesis of cadmium selenide quantum dots
4/25	11	Experiment 6 Characterization of cadmium selenide quantum dots
5/2	12	Experiment 7 (Exp. 5 report due) Exploration of tetraphenylporphyrin complexes
5/11	13	Experiment 7 Exploration of tetraphenylporphyrin complexes
5/16	14	Experiment 7 Exploration of tetraphenylporphyrin complexes
5/29		Writeup for Exp. 7 due

Detailed Description of Each Laboratory Experiment:

1. The Synthesis of Silly Putty, a Cross-linked Silicone Polymer
   :
   In this one-week experiment you will use trifunctional boric acid, $B(OH)_3$, as a cross-linking agent to synthesize a gummy material known as “silly putty”. Once the synthetic procedure has been carried out you will investigate some of the unique physical properties of this resinous material.
   :**Instrumentation used:** none
2. Synthesis, isomerization and ligand substitution of dichlorobis(ethylenediamine)cobalt(III) chloride
   : In this three-week experiment you will synthesize *trans*-dichlorobis(ethylenediamine)cobalt(III) chloride. Then, using two alternate oxidizing agents (hydrogen peroxide (H_2O_2) and oxygen (O_2)), you will isomerize the *trans*-$[Co(en)_2Cl_2]Cl$ to its *cis*-isomer. Finally, you will perform a ligand substitution reaction in which you will replace one of the Cl atoms on $Co(en)_2Cl_2$ with a sulfite ion to generate *cis*-$[Co(en)_2(SO_3)Cl]$. In addition, you will fully characterize all three products.
   :**Instrumentation used:** Infrared spectroscopy (IR), Ultraviolet-visible spectroscopy (UV-Vis), proton nuclear magnetic resonance spectroscopy (^{1}H-NMR).
3. Exploring the redox chemistry of chromium through the synthesis and reaction of chromium(II) acetate
   : In this two-week experiment you will synthesize and characterize chromous acetate, chromic sulfate and potassium chromate (using UV-Vis), and then react each with hydrogen peroxide under acidic and basic conditions and observe the oxidation state changes that occur for the chromium ion.
   :**Instrumentation used:** UV-Vis
4. Exploring the spectrochemical series through the synthesis and isolation of a series of chromium(III) complexes
   : In this two-week experiment you will synthesize a series of chromium(III) complexes (tris(ethylenediamine)chromium(III) chloride, potassium tris(oxalato)chromate(III) and hexakis(urea)chromium(III) chloride), then quantify Δ_o

by electronic absorption spectroscopy (specifically UV-Visible spectroscopy) and relate this to color and stability.

:**Instrumentation used:** UV-Vis

5. Preparation of tetrabutylammonium octachlorodirhenate

   : In this one-week experiment you will synthesize and characterize the first-known stable compound with a quadruple bond, $[Re_2Cl_8]^{2-}$. This lab is intended to expose you to oxygen-free synthetic laboratory techniques on the microscale and expose you to the transition metal rhenium for the first time.

   :**Instrumentation used:** UV-Vis, IR

6. Synthesis of CdSe quantum dots

   : In this two-week experiment you will synthesize and fully characterize CdSe quantum dots. This lab introduces you to the novel spectroscopic behavior of quantum-confined systems.

   :**Instrumentation used:** UV-Vis, IR

7. Exploration of tetraphenylporphyrin complexes

   : The final experiment for the semester is designed to develop your independent research skills. You will be tasked with researching and preparing one or more metalloporphyrin complexes and completely characterizing each step of the synthesis.

   :**Instrumentation used:** Methods chosen by the student