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Inorganic Chemistry Laboratory
CHM 3610L
Fall 2017

Lab
Friday 9:00 - 11:50 am
Building 50, Room 3608
Office Hours
MW 2-3pm, F 7:30-9am, 2-3:30pm

Course Information and Requirements

Required Materials:

In pursuit of the objectives outlined above, the course will utilize the following:

Lab Resources: Handouts will be used for the laboratory course. View the course website for a list of reference textbooks and resources.

Course Web Site:

http://www.unf.edu/~michael.lufaso/chem3610L/ and http://canvas.unf.edu/

Class Format:

The laboratory session will begin promptly at 9:00 am and end near 11:50 am. It is assumed all students will be in laboratory on time. You should arrive at the laboratory ahead of time to avoid traffic or other delays. At the beginning of each experiment I will discuss the theory behind the experiment and any pertinent safety precautions. Additionally, minimization of risks requires that students arrive well prepared for each lab experiment and are attentive. Because of the importance to you and your labmate's safety, it is *vital* that you be present for this discussion. For every minute late past the 9 am start time, your lab report will be reduced by 1% of the possible points, to a maximum penalty of a zero score for the lab report. If you are more than 50 minutes late, then you will not be allowed to perform the experiment or write the lab report and will receive a zero score. There will be no make-up experiments. Students are responsible for everything that is covered during the prelab discussion period and are advised to take detailed notes.

Disability Resource Center:

Students with disabilities who seek reasonable accommodations in the classroom or other aspects of performing their coursework must first register with the UNF Disability Resource Center (DRC) located in Building 57, Room 1500. DRC staff members work with students to obtain required documentation of disability and to identify appropriate accommodations as required by applicable disability laws including the Americans with Disabilities Act (ADA). After receiving all necessary documentation, the DRC staff determines whether a student qualifies for services with the DRC and if so, the accommodations the student requires will be provided. DRC staff then prepares a letter for the student to provide faculty advising them of approved accommodations. For further information, contact the DRC by phone (904) 620-2769, e-mail drcexams@unf.edu, or visit the DRC website www.unf.edu/drc

Military and veteran students may need both physical and academic accommodations and may contact the DRC to find further information. Military and veteran students who return from combat exposure may be utilizing the post 9/11 GI bill to continue postsecondary education goals. Contact Military and Veterans Resource Center by phone (904) 620-2655 or e-mail mvrc@unf.edu.

Pre-Labs:

A cursory understanding of the laboratory experiment is necessary for a safe working environment. The pre-lab must be completed prior to entering the laboratory. Upon entry, immediately place your notebook on the front desk. Before starting the experiment, I must approve your pre-lab in your laboratory notebook. If the pre-lab is incomplete or poorly written, up to 5% will be deducted from the possible score on your lab report.

Safety:

Safety is the number one priority in the inorganic chemistry laboratory. Students must display a serious attitude about learning and adopting safe laboratory practices. Casual or cavalier attitudes toward safety will not be tolerated, because such attitudes endanger your safety and the safety of those around you. The potential of risk is present in laboratory experiments. Care has been taken to minimize potential hazards. Although an accident is rare, it may happen. If an accident occurs, notify your instructor immediately. Performing unauthorized laboratory experiments is firmly prohibited. Unauthorized experiments present unacceptable and unknown safety hazards. The offense may be punished with a failing grade for that experiment or in extreme circumstances, removal from the course.

Food and drink are forbidden in the laboratory at all times. The use of headphones, cell phones, PDA, or laptops is not allowed in the laboratory. Those with cell phones that ring during lab may receive a penalty, per occurrence, to their overall course score. Students who don't bring safety goggles or are not wearing closed-toed shoes will be asked to leave. Either long pants to the ankle or a lab coat must be worn. *Goggles or safety glasses must be worn in the laboratory at all times*. The first offense, however slight or temporary, will result in a 3% reduction in the final score for the lab report. A second offense may result in dismissal from the laboratory for that day and a zero for the lab report. Shorts or short pants are not recommended and although a laboratory coat is not required, it is recommended. Open toed shoes are not allowed in the laboratory. When in doubt about any situation in the lab, consult with your instructor. In addition to these safety guidelines, there are also experiment-specific safety considerations. In the event of an evacuation (*e.g.* fire alarm) know how to evacuate and where to assemble.

Policy on Academic Integrity:

Any material submitted in the Inorganic Chemistry Laboratory must represent your own work and follow the Academic Integrity Code. Copying sections of the lab reports of others, including reports found on the WWW or those of previous students may be regarded as a violation of academic standards. A zero tolerance policy will be in effect. If you haven't already done so, you should familiarize yourself with UNF's academic policies and regulations, especially those dealing with academic integrity. The UNF undergraduate catalog http://www.unf.edu/catalog/ and the student handbook http://www.unf.edu/student-affairs/student-handbook.html contain more details regarding the Academic Integrity Code and possible faculty actions in a case of suspected academic misconduct.

Schedule:

The schedule may change as necessary as equipment needs and other scheduling considerations dictate. You will be informed, a week in advance, which experiment will be conducted the following week.

Fall 2017 schedule of inorganic laboratory experiments (tentative and subject to change).

	Day	
1	Aug 25	Syllabus, check in, safety, lab. notebook, instrumentation & equip. overview
2	Sep 1	Synthesis of $[Co(NH_3)_4CO_3]NO_3$
3	Sep 8	Synthesis of $[Co(NH_3)_5Cl]Cl_2$
4	Sep 15	Characterization of $[Co(NH_3)_4CO_3]NO_3$ and $[Co(NH_3)_5Cl]Cl_2$
5	Sep 22	Synthesis and characterization of linkage isomers: $[Co(NH_3)_5ONO]Cl_2$ and $[Co(NH_3)_5NO_2]Cl_2$
6	Sept 29	Synthesis, characterization, and resolution of enantiomers of $[Co(en)_3]^{3+}$, pt 1
7	Oct 6	Synthesis, characterization, and resolution of enantiomers of $[Co(en)_3]^{3+}$, pt 2
8	Oct 13	Preparation of an $[Ru(bpy)_3](BF_4)_2$ LED, pt 1
9	Oct 20	Preparation of an $[Ru(bpy)_3](BF_4)_2$ LED, pt 2
10	Oct 27	Synthesis of an air sensitive compound: copper(I) chloride
11	Nov 3	Synthesis and characterization of a high temperature superconductor: $YBa_2Cu_3O_{7-x}$
12	Nov 10	*Veteran's Day*
13	Nov 17	Synthesis and characterization of a high temperature superconductor: $YBa_2Cu_3O_{7-x}$
14	Nov 24	*Thanksgiving*
15	Dec 1	Lab checkout, exam/presentations

Grading:

Formal lab reports will be collected and graded, totaling 90% of the score. The final and lab safety is 10% of the score. Lab reports are due by noon on Monday ~10 days after the completion of the last lab in a series. A printed *and* electronic copy via flash drive or e-mail is required. Late lab reports will be penalized 3% per hour late. Grading will be based not only on the science, but also on clarity and writing skills. Lab safety and skill score is part of the lab report score and depends on how prepared and punctual you are, how well you use your lab time, your lab safety, lab awareness, and overall helpfulness, and the quality of your ideas, suggestions, and questions. The course will be graded so that A: 92 - 100%; A-: 90 – 92%; B+: 88 - 90%; B: 82 – 88; B-: 80-82; C+: 78-80%; C: 70 – 78%; D: 60 - 70%. +/- final grades will be assigned.

Student Learning Outcomes:

Content

- Demonstrate the ability to apply basic mathematics (arithmetic and algebra) and basic chemical principles to find solutions to simple quantitative problems and situations.
- Enhance and broaden your repertoire of synthesis and characterization techniques as related to inorganic chemistry.
- Perform laboratory experiments that are based on fundamental chemical principles and adhere to departmental standards of laboratory safety.

Critical Thinking

- Apply logical thought processes and background knowledge to draw appropriate conclusions from chemically related information and data.
- Infer conclusions and consequences from experimental data.

Communication

- Write professional papers in a style consistent with currently accepted scientific report structure to include proper grammar and spelling.

Laboratory Procedures:

Obtaining reagents: Balances must remain free of any visible trace of chemicals. A weighing dish should be labeled with your name and reagent prior to use. In order to avoid problems with the weighing area being in disarray, I reserve the right to *observe* and initial the entry in the lab notebook each weighing until proper techniques are established. A 1% penalty per occurrence will be in place for missing data. If chemicals are spilled and not immediately cleaned up, or reagent bottle tops are not replaced and closed, or a used weighing dish that is no longer needed is left on the benchtop then a 2% lab report penalty per occurrence may be enforced.

Labels: All glassware (i.e. beakers and flasks) containing any chemicals, including H_2O, must be labeled with: contents and initials. Unlabeled glassware containing a chemical will be confiscated, then returned with a 1% penalty to the student-owner that identifies their item.

Laboratory technique: Unsafe and poor laboratory technique is to be avoided. I will point out issues as they arise (e.g. evaluate and grade your weighing technique, apparatus setup, etc.). Repeated warnings, for poor technique on the same issue, will result in a penalty ranging from 1-3% reduction (per violation) in the possible score for the lab report, depending on the severity of the technique violation. Always leave the lab cleaner than you found it.

Laboratory notebook: A bound notebook is required to record all experimental data, procedures, and results. Recording data on scrap paper in not acceptable and the scrap paper is subject to immediate *recycling*. Data not immediately recorded, e.g. at the balance, may result in a penalty of 1% for the lab report. A lack of record of procedures that were performed and observations not clearly and timely recorded, may result in a penalty of 1%, per occurrence.

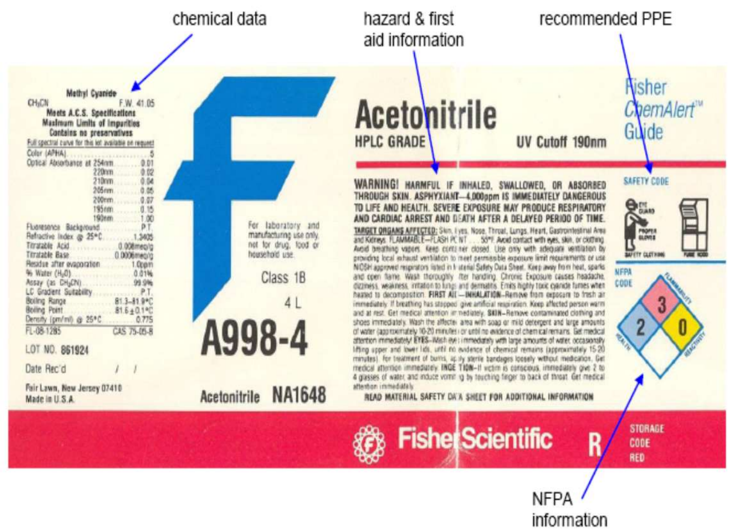
Spectroscopic Instrumentation: The UV-Vis spectrometer and IR spectrometer are expensive and fragile instruments and must be treated as such. Careless use of the instruments will not be tolerated. In laboratory experiments where the use of IR for UV-Vis spectroscopy is required, I reserve the right to accompany each person to the room to individually use the instrumentation until proper use of the instrumentation by each student can be established. A sign-in sheet may be utilized. All chemicals must be immediately cleaned from the instrumentation area with a Kimwipe or equivalent. Leaving the instrumentation area in an unclean state will result in up to a 5% reduction in the possible score for the lab report.

Fume hood, bench area: These areas must be clean at the end of the laboratory. Visible evidence of chemicals or waste will result in a penalty ranging from 1-10% reduction in the possible score for the lab report, depending on the severity of the chemical waste area.

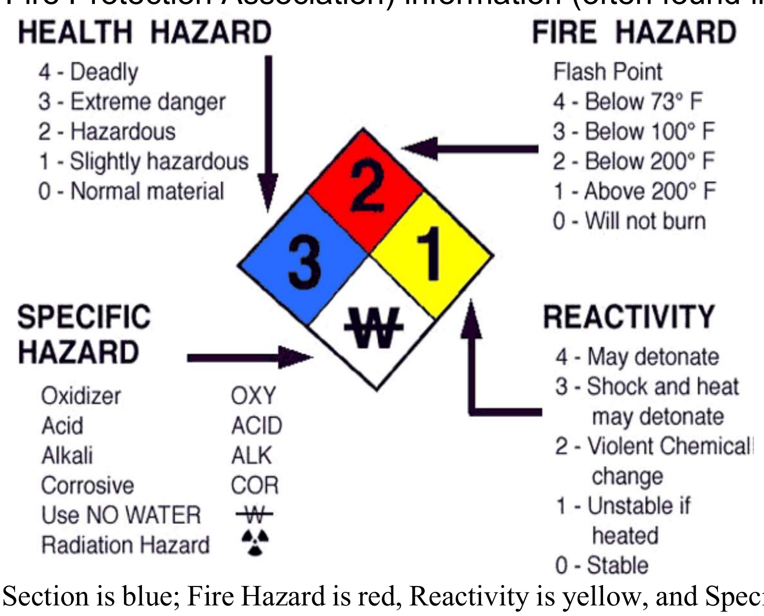
When using chemicals:

- ***Read the label carefully***
- ***Understand the potential hazards*** of all chemicals before using them
- ***Never taste*** a chemical or solution
- ***Avoid inhaling vapors*** and keep open vessels containing organic solvents in a hood at all times
- Dilute acid by adding ***acid to water***. *Never add water to concentrated acid*
- Keep reagent bottles tightly capped when not in use

The label on the chemical bottle is the easiest thing to check. Labels do not provide in-depth quantitative information, but will usually tell you the types and levels of hazard posed by a chemical.



NFPA (National Fire Protection Association) information (often found in a diamond):



The Health Hazard Section is blue; Fire Hazard is red, Reactivity is yellow, and Specific Hazard is white. For the first three sections, in each case they are rated from 0-4, with 4 indicating the highest hazard level. For more information about the chemical, its hazards, proper handling and emergency response, use the MSDS. MSDS: Material Safety Data Sheets provide more information about chemical toxicity. The content and quality of MSDSs varies with the manufacturer, so it may be worthwhile to look at multiple sheets. MSDS's are available on-line (www.hazard.com).

Chemical Spill Prevention & Waste Collection

Spill Prevention
At all times you should work carefully to avoid spills. This means:

- Good Housekeeping: you are less likely to tip something over when your area is neat
- Keep lids on reagent bottles when not in use
- Flasks with liquids should be clamped or supported to prevent tipping. A thermometer in a flask should be clamped rather than just set in the flask. A thermometer (or stirring rod or spatula) left sticking out of a flask will increase the chances of it tipping.

Spills and Splashes
If a chemical contacts your skin, rinse the area with water. If necessary, use the safety shower. Any clothing saturated with chemicals must be removed; this is not a time for modesty.
If you are splashed in the face but have splash-proof goggles on, use the eye wash, *leaving your goggles on* so that the chemicals do not get rinsed into your eyes.
If you are splashed directly in the eyes, use the eye wash. For maximum effectiveness you must have your eyes *open*; use your fingers to hold them open if necessary.

Waste Collection
Because different institutional disposal methods are required for different classes of chemicals, we will always collect our chemical waste into different labeled bottles. Always place waste in the appropriate waste bottle, for your safety and for the sake of the environment. Waste bottle types in our labs are:

- Aqueous Waste: While some aqueous waste can go down the drain, most of the aqueous waste generated in the experiments must be collected in waste containers. Use the sinks in the fume hood if you are informed that waste is okay to go down a drain. This will serve to minimize laboratory odors.
- Non-Halogenated Organic Waste: compounds which do NOT contain a carbon-halogen (fluorine, chlorine, bromine, iodine) bond.
- Halogenated Organic Waste: compounds which contain a carbon-halogen bond.
- Heavy metals: Solids or solutions containing heavy metals are often collected separately so that the heavy metals can more readily be recovered.
- Toxic or Reactive Substances: If substances are particularly toxic or reactive, they may be collected separately to insure safe handling.

Waste bottles should not be overfilled. Pay attention to the fill level of waste bottles. If a bottle is getting full, notify the instructor so that a new waste bottle can be provided.

Please remember to fill out the waste label, full name of chemicals, when you add to a new waste bottle.

Safety Equipment

Introduction
Fire is the most common serious hazard that one faces in a typical chemistry laboratory. While proper procedure and training can minimize the chances of an accidental fire, you must still be prepared to deal with a fire emergency should it occur. You should NEVER attempt to fight a fire.
Fire extinguisher types, as well as the proper procedures to follow should a fire occur, are described below. It is **not** a comprehensive guide.

Stop, Drop, and Roll
If your clothing is on fire STOP, DROP and ROLL on the ground to extinguish the flames. If you are within a few feet of a safety shower, you can use these instead. If one of your labmates catches fire and runs out of the lab in a panic, tackle them and extinguish their clothing.

Fire Classification
The National Fire Protection Association (NFPA) classifies fires into five general categories (U.S.):

- **Class A** fires are ash-producing materials like burning paper, lumber, cardboard, plastics etc.
- **Class B** fires involve flammable or combustible liquids such as gasoline, kerosene, and common organic solvents used in the laboratory.
- **Class C** fires involve energized electrical equipment, such as appliances, switches, panel boxes, power tools, hot plates and stirrers. Water can be a dangerous extinguishing medium for class C fires because of the risk of electrical shock unless a specialized water mist extinguisher is used.
- **Class D** fires involve combustible metals, such as magnesium, titanium, potassium and sodium as well as pyrophoric organometallic reagents such as alkyllithiums, Grignards and diethylzinc. These materials burn at high temperatures and will react violently with water, air, and/or other chemicals. Handle with care!!
- **Class K** fires are kitchen fires. This class was added to the NFPA portable extinguishers Standard 10 in 1998. Kitchen extinguishers installed before June 30, 1998 are "grandfathered" into the standard.

Some fires may be a combination of these! Our fire extinguishers have ABC ratings on them. These ratings are determined under ANSI/UL Standard 711 and look something like "3-A:40-B:C". Higher numbers mean more firefighting power. In this example, the extinguisher has a good firefighting capacity for Class A, B and C fires. NFPA has a brief description of UL 711 if you want to know more.

Basic Types of Fire Extinguishers
The two most common types of extinguishers in laboratories are pressurized dry chemical (Type BC or ABC) and carbon dioxide (CO_2) extinguishers:

Response to a Fire
You should NEVER attempt to fight a fire. However, in the event of a fire, you should respond in the following manner.

1. Shout to notify your instructor that there is a fire.
2. Your instructor will indicate whether evacuation is necessary.
3. If we evacuate, we will:
    - Exit the building calmly via a stairwell that is the opposite direction of the fire
    - Assemble in the center area between buildings 50 (Science and Engineering) and 4.
    - Pull the fire alarm on the way out.
4. If necessary, be sure to stay low and avoid smoke.

Lab Safety Officer

Each student will be assigned to act as a laboratory safety officer for at least one week during the semester. The presented will research the hazards of that lab and ways to minimize the associated risks, and make a brief (~ 2-4 minute) pre-lab presentation to the class. The laboratory safety officer will act as a monitor, during the lab, for good laboratory practices and will be responsible for ensuring that the lab is left in good order.

Pre-lab Presentation
In your pre-lab safety presentation you will explain to the class the four major categories of safety issues for that day's experiment:

I. Chemical Hazards

II. Procedural Hazards

III. Personal Protective Equipment (PPE)

IV. Waste Disposal

Using your lab manual generate a list of all chemicals that will be used and produced during your assigned experiment. Use MSDSs (available in the laboratory or online at www.hazard.com or similar website) to learn about proper handling of the chemicals. What are the hazards associated with these chemicals? What sort of PPE (personal protective equipment) should be used when handling them? What wastes will be generated, and how should they be disposed—in the aqueous waste bottle, in the non-halogenated organic waste container, in the halogenated organic waste container, in the trash?

Assessing Laboratory Practices
During the lab, the safety officer will monitor the class for poor laboratory practices such as failure to use PPE, eating or drinking, leaving reagent bottles uncapped, failure to clean up spilled chemicals in the fume hood or near the balances, or improper disposal of chemical waste. Good laboratory practices are everyone's responsibility; during your assigned week it will be your job to raise awareness of ways in which your labmates (and instructor) could improve their laboratory practices. The safety officer must be present for the entire lab period.

Post-lab Inspection
During your assigned week, the safety officer will be responsible for conducting a post-lab inspection of the laboratory. The safety officer must be present for this inspection. Remedy any problems you identify (waste improperly disposed, trash on the benches, etc.) with the help of your instructor.

Scoring
You will receive an individual score for your performance. The score will be based on your pre-lab presentation and leadership during the lab period, completion of the post-laboratory inspection, and the state of the laboratory when you leave.