Chemistry 145 Lab – Spring 2021

Inorganic and Organometallic Chemistry with Laboratory

Prof. Wan-Yi “Amy” Chu (she/her/hers)

Email: [amchu@mills.edu](mailto:amchu@mills.edu)

* **Point distribution**

|  |  |
| --- | --- |
| Pre-lab discussion | 50 points |
| Lab notebook | 150 points |
| Total | 200 points |

* **Lab Schedule: Spring 2021**

|  |  |  |
| --- | --- | --- |
| **Date** | **Location** |  |
| 1/22 | Online Zoom | In-person lab logistics and introduction to semester project |
| 1/29 | NSB 201 | Exp. 1 Redox agents: synthesis of ferrocenium tetrafluoroborate |
| 2/5 | NSB 201 | Exp. 2 Dissolving metal reduction: synthesis of dppe in ammonia - 1 |
| 2/12 | NSB 201 | Exp. 2 Dissolving metal reduction: synthesis of dppe in ammonia - 2 |
| 2/19 | NSB 201 | Exp. 3 Weakly coordinating ions: synthesis of NaBArF4 - 1 |
| 2/26 | NSB 201 | Exp. 3 Weakly coordinating ions: synthesis of NaBArF4 - 2 |
| 3/5 | NSB 201 | Exp. 4 Synthesis of DMSO complexes and IR spectroscopy |
| 3/12 | NSB 201 | Exp. 5 Thermal isomerization of [Co(en)2Cl2]Cl |
| 3/19 | NSB 201 | Exp. 6 Synthesis and magnetic moment of copper acetate |
| 3/26 | *Spring break, no lab* | |
| 4/2 | NSB 201 | Exp. 7: Spectrochemical series: synthesis of octahedral cobalt complexes |
| 4/9 | NSB 201 | Exp. 8: Synthesis of a model coordination complex of vitamin B12 |
| 4/16 | NSB 201 | Exp. 9: Synthesis and catalytic activity of Wilkinson’s catalyst |
| 4/23 | Online Zoom | Peer review of 1st draft of slide deck |

* **Introduction**

This is an inorganic synthesis lab. It is a hands-on intensive laboratory experience that require a lot of preparative work and attention. The focus of lab is to give you experience in practical inorganic synthetic and analytical techniques. Because we are mostly focused on practical skills, the objectives of the experiments may not have intimate connections to the concepts that we discuss in lecture. Thus, you must prepare before each lab by thoroughly reading the introduction and prepare answers to the study questions.

* **Required materials:**

1. Bound laboratory notebook. Your lab grade is mostly based on what you write in your laboratory notebook. You must have a permanently bound notebook to record your lab work that will be used for grading.
2. Lab coat for working in the lab. Must completely cover your arms and upper body.
3. Lab goggles or lab glasses. If you do not have one you can borrow one from the instructor. **You are in charge of sanitizing it afterwards.**
4. A signed copy of the “Summary of Safety Rules” document, submitted to the instructor at the beginning of the first lab.

* **Pre-lab discussion:**

Before each experiment, we will discuss the main concepts and techniques in that experiment. I will ask questions from the **Study Problems** listed in each experiment before the procedure. For each response that you give you will earn points. You can answer a question or add on to another student’s response. This accounts for 50 pts of your total grade.

The best way to prepare is to read the lab manual before coming to lab and have a set of answers prepared for the study problems. You can also discuss with your classmates, even divide up the questions so that everyone can reach 50 pts together at the end of the semester!

Pre-lab discussion rubric:

|  |  |
| --- | --- |
|  | points |
| Highly quality response | **8** |
| Medium quality response | **5** |
| Low quality response | **2** |
| Response to another classmate that add quality to the original answer | **5** |

* **Lab notebook:**

Notebook records are due 1 week after the experiment has concluded. Typically, students will finish writing the lab notebook before leaving class. It doesn’t take a lot of time if your memory is fresh! Scan all your pages and upload to Canvas as a pdf.

Your notebook should be permanently bound, and each page should be numbered and have your name and the date. There should also be a table of contents at the beginning of the notebook. You must write in ink in your notebook, and you should record all observations and data directly into your notebook. If you write in pencil or make notes on pieces of paper that are not in your notebook, I will take a point from your total notebook score.

There are 9 labs in this course. Notebook records for each lab is worth 15 points each except for labs 2 and 3 which are worth 30 points each. I will drop the lowest grade lab record from your final grade. Your records must be neat, well-organized, written in ink, with a table of contents and the other required elements. If you need to change anything in your notebook, line out the old information neatly with a single line. Do not overwrite or scribble out old information. I should easily be able to find information in your notebook. If I cannot, it will impact your grade for organization and neatness.

Your notebook should have the following elements:

Objective:

Complete before coming to lab. This should be a 1 – 2 sentence description of the experiment. Since this is a synthetic technique lab, your objective is usually “to synthesize \_\_\_\_\_\_\_\_\_ using \_\_\_\_\_\_\_\_\_\_ and analysis with \_\_\_\_\_\_\_\_\_\_\_”. State the product, important reagents or synthetic techniques, and analysis method.

Reaction equations:

Complete before coming to lab. You must write every reaction equation in your notebook with the complete structure for every reactant, reagent, and product drawn. DO NOT write the equation using formulas.

Chemical information:

Complete before coming to lab. Include the structures, molar masses, boiling points, melting points, and densities (of liquids only) of all reactants, reagents, solvent, and products.

Safety disclaimer:

Complete before coming to lab. If there are any chemicals used in the experiment that have particular hazards (toxic, explosion risk, corrosive….etc.) you must write a safety note.

Procedure and observations:

This part is graded on completeness. The more detailed the higher the score. A complete documentation of what happens during the experiment. Record all masses, volumes, glassware used. If there are unusual glassware setups you must draw it in your notebook. Record all color changes or any other changes to appearances.

Spectra:

Append the spectra from analysis. For experiments that do not have spectra you will automatically receive full marks for this portion.

Calculations:

Include any calculations of stoichiometry, yield, or measurements.

Conclusions:

Include all yields and analysis of spectra.

In upper division labs, which have only one session per week, you must attend every lab period. There are no possibilities for lab make ups. If you miss a lab, you automatically lose all the points for the “Procedure and observations” and “Calculations” category, since those records can only be obtained if you were physically in lab. I will drop the lowest grade lab record from your final grade.

Notebook rubric

|  |  |
| --- | --- |
|  | points |
| Objective | **/1** |
| Reaction equations | **/1** |
| Chemical information | **/2** |
| Safety disclaimer | **/2** |
| Procedure and observations | **/4** |
| Spectra | **/1** |
| Calculations | **/1** |
| Conclusion (analysis of yield and spectra) | **/3** |
| Total for each experiment | **/15** |
| \*Points in each category are doubled for Exp. 2 and 3 | |

**Summary of Safety Rules**

**Safety against infections viral diseases**

1) You must wear a mask in lab at all times. The mask must fully cover your mouth and nose.

2) Staying within 6 ft of others for more than 10 mins is prohibited. You must stay in your assigned work bay during all times.

3) If you are showing symptoms of a viral infection, you must not enter the lab.

4) In addition to the above protocols, you must abide by general CDC guidelines on limiting the spread of COVID-19 whenever you are on campus.

**Personal Safety**

1) You must know the locations of the safety shower, eye wash, fire extinguishers and exits.

2) In case of fire call the instructor at once and alert others.

3) In case of fire or earthquake, leave the building according to the evacuation plan as quickly and safely as possible, but do not panic.

4) Eye protection must be worn at all times.

5) Never eat, drink or smoke anything in the laboratory. Never taste a chemical.

6) Never pipet by mouth.

7) Do not wear sandals in the laboratory. Tie back long hair and loose clothing to prevent spills and other mishaps. Gloves and aprons are provided for your safety.

8) Work neatly. Turn off equipment and water taps when not in use and throw away used paper towels.

9) If you feel faint or unwell while working in the lab, inform your instructor and leave the laboratory.

10) Never work in the laboratory without permission of the instructor. Perform only authorized experiments.

11) Never work alone in the laboratory.

12) Do not use any flame in the laboratory. All heating will be done with hot plates and water, oil, or sand.

13) When boiling liquids you must use agitation to prevent bumping. If you forget to do this, never add agitation to a hot liquid.

14) Be aware of the properties and hazards of the chemicals you are working with. Take extra precautions as needed.

15) Examine all glassware and equipment for defects before use. If broken or cracked, replace the item.

16) Always wash your hands before leaving the laboratory.

17) Students who are pregnant or nursing must make arrangements to take the lab at a later time.

**Breakage of glassware**

1) Never heat a closed vessel.

2) Never use pressure on glassware, it does not bend at room temperature. This is especially important for the insertion of glass tubing and thermometers into adapters. Open the adapter, insert the thermometer and tighten.

3) Lubricate glass joints or they will get stuck. If a joint is stuck, ask for help.

4) Sweep up all broken glass immediately and place in proper container.

**Use of Chemicals**

1) Always wear gloves! Do not touch chemicals unnecessarily. Transfer solids with spatulas and liquids by pouring or with pipets.

2) Never taste chemicals in the lab. If you want to smell a chemical, keep it at a safe distance from you and use your hand fan the smell towards you.

3) Do not use any unknown or unlabeled chemicals, unless given to you as "unknowns" to identify.

4) Read labels carefully to make sure you use the correct reagents.

5) Never return excess reagent to the bottle. Share your excess with another student or dispose of the chemical correctly.

6) Avoid breathing fumes such as noxious gases or solvent vapors, by using a gas trap or by working in the hood.

7) Pour acids into water, never water into acids.

8) If a chemical spill occurs, ask for help immediately.

9) Many common chemicals are suspected of being carcinogens. If no substitute is available, we may use them in the lab. At that point you must strictly avoid contact by wearing gloves and/or working in the hood.

10) Many chemicals are classified as mutagens (positive Ames test with bacteria). Most of these are also carcinogens and so they should be treated as such.

**Disposal of Chemicals**

1) Only non-toxic, aqueous or water soluble liquids may be discarded in the hood

sinks.

2) Non-toxic solids may be placed in the appropriate waste container.

3) Toxic substances and organic liquids are to be discarded in the appropriate waste container in the hood.

4) If you are unsure of where or how to dispose of any substance, ask your instructor or the stockroom supervisor for assistance.

**Accidents**

1) Move to safety, then call the instructor or assistant.

2) Report all spills and injuries to instructor immediately.

3) Clean up minor spills at once.

4) Whenever you spill a chemical on yourself or burn yourself, rinse the af­fected area with copious amounts of cool water at the main sink.

4) In the event of a fire, alert everyone and run outside.

5) In the case of a major accident, call the emergency number (x911) and tell them that there is an accident in NBS-201. Then call PUBLIC SAFETY, x5555.

*Read this list carefully and return a signed copy of this form before beginning lab work. If you have any questions, please ask before signing.*