

### GENERAL INFORMATION

Instructor: David Glueck (305 Burke)

Teaching Assistants: Brian Anderson (318 Burke)  
Jian Yuan (310 Burke)

Time: Thursday or Friday, 2:00-6:00 PM, 306 Steele

Laboratory Notebook: this will be issued when you check into the lab (do not purchase one at the bookstore).

Schedule: Your lab manual contains six experiments (see the brief descriptions on the next page). Each laboratory period will begin with some comments in the laboratory about the experiment; **please do not be late for the start of lab**. Experiment 1 is a "dry lab" and will require no lab report.

**Lab reports for the remaining experiments will be due within one week of your lab section.** (Thus, if you have lab on Thursday January 17, your report is due on Thursday January 24). You can turn in reports to the TA's or to me. Please don't pester the TA's with questions about the lab reports; ask me instead.

**There will be a penalty for late laboratory reports!**

- one or two days late -- 20% penalty. Example: you're 2 days late and get a score of 8 out of 10. After deducting 20% your corrected score is 6.4 out of 10.
- 3 to 6 days late -- 40% penalty. Example: you're 5 days late and get a score of 6 out of 10. After deducting 40% your corrected score is 3.6 out of 10.
- more than 6 days late -- 60% penalty. Example, you're 2 weeks late and get a score of 4 out of 10. After deducting 60% your corrected score is 1.6 out of 10.

Because of possible delays between doing the experiment and writing up the lab report, accurate and complete observations in your **notebook** are crucial. This is especially important since part of your lab grade (see below) will be based on evaluation of your notebook.

Fees: The standard laboratory charge, which includes the cost of nonreturnable items and the laboratory notebook, is **\$16.00**. Further, additional charges will be made for breakage, and/or excessive damage and the total sum will be added to your College bill at the end of the term. Please take good care of your equipment--it is expensive.

Grading: Your lab grade is based on:

Reports	70%
Notebooks	20%
Instructor's Evaluation of Technique and Comprehension	10%

**The laboratory portion of the course will count for about 25% of the final grade.**

**However, failure to complete the laboratory (this includes turning in the lab reports!) will result in a failing grade.**

## LABORATORY EXPERIMENTS

1. **Molecular Structure and Symmetry (1/12 - 1/13):** Apply VSEPR to build models of molecules and assign them to their molecular symmetry groups (point groups).
2. **Spectra and Structure of Nitrile Complexes (1/19 - 1/20):** Prepare a copper acetonitrile complex and a palladium benzonitrile complex. Use IR and NMR spectra to determine their structures.
3. **Synthesis and Resolution of a Chiral Nickel Complex (1/26 - 1/27):** Prepare and separate a racemic mixture of Ni-phenanthroline complexes. Study the chiroptical properties of one enantiomer of the Ni complex.
4. **Four-Coordinate Nickel Diphosphine Complexes (2/2 - 2/3):** Prepare two closely related diphosphine complexes of nickel dichloride, and compare their physical and spectroscopic properties.

5. No lab this week, *Winter Carnival holiday Friday 2/10!*
6. **Stabilization of Chromium(II) by Complexation (2/16 - 2/17):** Use inert atmosphere techniques to prepare air sensitive solutions of Cr(II). Stabilize this oxidation state by ligand coordination and make a compound with a metal-metal multiple bond.
7. **Preparation of Chromium(III) Compounds: the Spectrochemical Series (2/23 - 2/24):** Prepare Cr(III) compounds by three different procedures, in one case using liquid ammonia, and study their electronic properties that lead to their distinctive colors spectroscopically.

# ACADEMIC HONOR PRINCIPLE

The principle of academic honesty is at the very heart of experimental science. The following remarks apply to the laboratory work in Chemistry 64:

- Unless permission is granted by the instructor, use of another student's laboratory data is a violation.
- When use of another's data is allowed, the source of the data must be indicated.
- Fabrication of data or alteration of your own data to secure some desired result is also a violation. *[This happened in Winter 2001 and resulted in a 1-term suspension.]*
- In the case of experiments where two students work together and data have been recorded in one student's notebook, a copy of the data may be made in the other student's notebook with an appropriate citation to the location of the original data. Any other material in the notebook which has been copied from any source whatever must also be provided with a source citation.
- The laboratory report must represent your *independent* calculations and *individual* conclusions, although comparison of numerical results with another student is permitted, with appropriate attribution.
- Of course, direct copying of any portion of another student's laboratory report is a clear violation of the Honor Principle.

Any violation of the Honor Principle in this course will result in notification of the College Committee on Standards.

If you have ANY QUESTIONS about how the Honor Principle applies to any situation in this course, PLEASE DO NOT HESITATE TO CONTACT YOUR INSTRUCTOR.

## LABORATORY SAFETY

1. Wear safety goggles in the lab. Repeated violation of this rule will lead to ejection from the lab.
2. Never use an open flame in the lab.
3. Smoking, eating, and drinking is absolutely forbidden in this laboratory.
4. Never bring coats or bulky flammable items into the laboratory.
5. Know the location of the nearest fire extinguisher, safety shower, eye wash fountain, and exit.
6. Do all pipetting with a suction bulb. Mouth pipetting is absolutely forbidden.
7. When a reaction is left alone for extended periods of time ( $>1/2$  hour), leave a note next to it indicating important facts for persons working in that vicinity.
8. Know the physical, chemical, physiological properties (as far as possible) of the reactants, products, and solvents used in each preparation. Be ready for questions about the toxicity of the materials which you will be using.
9. Do all manipulations **in the hood** unless directed otherwise.
10. Know and follow the proper disposal method for chemicals.
11. Clean up your work area in the laboratory before you leave.
12. Wash your hands thoroughly before leaving the lab.
13. If you have any questions or doubts about the experimental procedures, contact the instructor or the teaching assistants **before** beginning experimental work.

## LABORATORY GUIDELINES

1. Safety glasses must be worn by everyone who enters the lab.
2. No sandals or shorts are to be worn while in the lab.
3. Do the experiments **in the hood**.
4. Cleanliness of the laboratory is the responsibility of the students who use it and the Teaching Assistants who supervise them.

At the conclusion of each lab session:

- All glassware must be cleaned and put in the lockers. **Do not put dirty glassware in the lockers.**
- Benchtops are to be cleaned of any spills and chemical residue.
- Balances and the areas around them are to be free of spilled chemicals and used weighing paper.
- Reagent dispensing areas are to be free of spilled chemicals or liquid reagents.
- Hoods are to be clean and emptied of all glassware and chemicals.

**The TAs will inspect your lab station and associated common equipment areas before signing your lab notebook. They will also provide brief qualitative assessment of your notebook and lab technique each week.**

6. Place all broken glassware in the SHARPS containers. **Do not put broken glass in the trash.**
7. Dispose of waste chemicals in the containers provided. **Do not put waste chemicals or products in the lockers.**
8. Report any broken glassware to the TA for replacement.

## LABORATORY REPORTS

In the laboratory, record **all** pertinent observations and data for each experiment in a neat, concise fashion **in pen in your notebook as you do the experiment**. Writing up a prelab as done in other courses is also useful. Notebooks may be collected, and examined, **without notice**, at various times during the term; evaluation of your notebook counts as part of your lab grade.

*Here is the format for the lab reports, which should be **BRIEF but clearly written**.*

**1. TITLE & DATE:** The title of the experiment and the date it was done should be listed on the front page along with the name(s) of your lab partners.

**2. INTRODUCTION:** (BRIEF!) Background material explaining why you are performing the experiment. Information for this section can be found in the lab handout and/or in the references given at the beginning of each experiment.

**3. RESULTS & DISCUSSION:** What happened and how you interpret it. Information to include here: what compounds you made, their spectral data, answers to the questions from the lab manual, and discussion of the results. If you use an idea from a reference to help you answer a question, the reference should be footnoted (include the references as endnotes at the end of the report).

**4. EXPERIMENTAL:** Each new complex isolated should be listed separately. A good format is:

Complex **aa**: A sample of \$\$ [X g, Y mol] was dissolved in H<sub>2</sub>O (30 mL) to afford a green solution which was then added to a blue solution of ## [X g, Y mol] in MeOH (10 mL). The solution was stirred at room temperature for one hour. Filtration of the resulting purple precipitate and subsequent washing with H<sub>2</sub>O (3 x 15 mL) yielded, before recrystallization, purple crystals of @@ (X g, Y mol, % yield), m.p. 102-104 °C.

**5. CONCLUSION:** This can be brief, but you need to summarize the results.

**6. REFERENCES:** Use the standard American Chemical Society format:

(1) Huheey, J. E. *Inorganic Chemistry: Principles of Structure and Reactivity*, 2nd Ed.; Harper & Row, New York, 1978, 498-507.

(2) Schlafer, H. L. *J. Phys. Chem.* **1965**, 69, 2201-2208.

All lab reports **MUST** be printed or typed (handwritten reports will not be accepted). Spectra should be stapled to the last page or pasted into the report itself.