

CH 402

Inorganic Chemistry Laboratory

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Objectives: This laboratory is intended to introduce the student to some of the more common experimental techniques and methods used by inorganic chemists. Another important goal is the review of methods and techniques which were encountered in earlier laboratory courses in chemistry. As in any laboratory course, principles and theories which are covered in lecture will be illustrated as well. A wide variety of synthetic procedures, methods of purification and characterization, and types of instrumentation will be used.

Grading: Grades are based on the following:

- Laboratory Reports (formal write up, laboratory notebook)
- Experimental Results (i.e., how much effort do you put into the lab)
- Techniques, safety practices, preparation, etc.

Glassware: Laboratory glassware is incredibly expensive. Note that a condenser costs about \$50. When experiments are carried out using glassware, some things will get broken and we expect that. However, excessive breakage, or breakage resulting from carelessness are unacceptable laboratory practices---poor laboratory technique, and will be graded accordingly. When you break glassware be sure to dispose of it correctly. For broken beakers, Erlenmeyer flasks, etc., that do not have ground glass joints, just dispose of the broken glass ***in the broken glass container, not just in a trash can***. For glassware with ground glass joints, the joints, if they themselves are not broken, should be salvaged. For flasks with star cracks, save the flask - it can be repaired by the glassblower. For broken thermometers, be sure to clean up the mercury mess as well as the glass.

Safety: Because flammable liquids and corrosive and health-damaging chemicals are frequently encountered in the chemical laboratory, unsafe laboratory practices cannot and will not be tolerated. Safety clothing: Safety glasses (or prescription glasses) are required at all times while in the laboratory. Rubber gloves are required while carrying out experimental work, handling chemicals, etc. A laboratory coat or apron is recommended but not required. Know where the fire extinguisher, safety shower, and eyewash fountain are located.

Chemical Waste Disposal: In order to avoid environmental contamination, laboratory chemicals must be disposed of properly. Non-halogenated solvents must be poured into the "non-chlorinated waste" jug; halogenated solvents are poured into the "chlorinated waste" jug. Solid chemical waste is placed in the

appropriate chemical waste bucket, broken glass is placed into the broken glass container. **Never put solids, organic wastes, or heavy-metal salts down the drain!**

Laboratory Notebooks: The notebook serves as a source of information for you to carry out the experiment **and** provides a record of your experiment, observations, etc. You have to record your work **as you have done it (third person, past tense)** in concise form but with sufficient clarity and detail so that a person of equivalent training and experience could repeat your experiment. This should constitute the basis of the **Experimental Section** of your formal write-up.

Laboratory Reports: Your formal write-up (typed, handwritten lab reports are not acceptable) should include the following:

Title of the Experiment

Abstract (a few sentences which summarize, in general terms, the experiment, reactions, procedure, and results).

Introduction (a few sentences which introduce the reader--the instructor--to the experiment).

Experimental Section (this should be a brief and to-the-point description of the experimental details; third person, past tense, passive voice; see an issue of the journal *Inorganic Chemistry* to see how an experimental section is written).

Results (a one or two sentence summary of the results of your experiment. Use a table whenever this will improve presentation).

Discussion It will be useful and necessary to interpret your experimental results in this **Discussion Section**. A Discussion Section should include chemical reactions and important reaction mechanisms. You need to indicate to the instructor that you fully understand all of the techniques and chemical concepts involved in the experiment. Often, you are asked to include study questions/exercises in your discussion and to draw some conclusions from your results. Incorporate these study questions into your discussion. Don't just have "naked" answers numerically listed. Note that a good **Discussion Section** will often require consulting additional literature sources (the *Inorganic Chemistry* textbook, the library, chemical journals). Be sure to adequately reference sources that you use in your writeup (that is, reference facts gathered or statements made from literature sources, use numerical superscripts in your writeups and compile the references at the end of your writeup; **don't just have a bibliography at the end and make me guess from which of these you got your information**).

References and Notes failure to adequately reference your sources does not do justice to those people on whom you are basing your write-up and may constitute plagiarism. **Give adequate references to all literature sources.** See a recent issue of the *Inorganic Chemistry* to see how references are handled, listed, and abbreviated. This section will be worth 10% of your write-up score.

CAUTION: DO NOT PLAGIARIZE TEXTBOOKS, JOURNAL ARTICLES, OR OTHER STUDENTS. USE YOUR OWN WORDS!!

Schedule: Each student is responsible for her/his own schedule. At the beginning of the semester you will meet with the instructor, provide a working e-mail address, and tell the instructor which day(s) of the week you can perform lab work. The instructor will then coordinate all schedules and let you know which day(s) of the week you can perform your experiments. There will be a total of six experiments:

1. Preparation of $[\text{Co}(\text{NH}_3)_4\text{CO}_3]\text{NO}_3$
2. Synthesis of (tetraphenylporphyrin)Ni
3. Preparation of Ferrocene
4. Preparation of (Mesitylene)Mo(CO)₃
5. Preparation of Chromium(II) Acetate Hydrate
6. Synthesis of Dibenzotetraaza[14]annulene

Your first assignment is to find peer-reviewed publications detailing the synthesis of these compounds. A list with the structures and chemical formulas of the compounds that assists in your search is supplied separately. The instructor will introduce you to modern methods of literature searches (such as SciFinder Scholar) on request. Once you found (a) publication(s) describing the synthesis, you will present this to the instructor, who in turn will provide you with an experimental describing the syntheses as you will perform them. **The internet is not peer-reviewed, therefore synthetic procedures found on the web will not be accepted.**

These experiments can be performed in any sequence the student chooses. Some of the experiments are multi-step synthesis, and might need more than 1 day to finish. Be aware of this (i.e., read the experimental before you start the experiment) and schedule accordingly. This is important because the instructor will perform occasional quizzes to establish your knowledge of the experiment you wish to perform. **If you are found to be insufficiently prepared for an experiment you will be sent home and lose that lab day!**

Once you have finished your experiment you have **one week** to turn in the lab report. In case you need NMR, IR, and or UV-vis spectroscopy to characterize the compound(s) you have prepared, let the instructor know **before you begin** the experiment so that he can schedule instrument time for you.