Spring 2011

CHEM-458 INORGANIC CHEMISTRY LABORATORY

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I. Overview

The inorganic chemistry laboratory is an introduction to the synthesis and characterization of inorganic compounds. As you will learn the diversity of molecular and electronic structures of inorganic compounds provides is rich. A wide range of physical methods must be brought to bear to fully characterize these materials.

The objectives of the laboratory course include:

a) the illustration of some of the principles of inorganic chemistry.
b) training in the techniques of inorganic chemical synthesis.
c) training in the presentation of experimental data and discussion of results.
d) training in the use of reference materials in the interpretation and discussion of results.
e) development of a research attitude and skill set.

The experiments have been selected to provide you with an opportunity to become familiar with synthetic techniques, to attain knowledge of some physical methods and their applications in solving inorganic problems, to gain some insight into correlating experimental observations and data with theory, and, in general, to help you develop an appreciation for and interest in inorganic chemistry and synthesis.

II. Organization

The five experiments to be performed this semester are but a few of the many which could have been chosen for this course. Inorganic chemistry has expanded rapidly to include a rather wide and diverse scope of topics, ranging from the traditional study of main group and transition metal coordination compounds, to the areas of organometallics and bioinorganic model systems. The experiments that will be conducted during this laboratory course are the following:

a) The Synthesis and Characterization of Some Coordination Compounds of Nickel – A study of coordination compounds and the determination of structure from magnetic susceptibilities and optical absorption spectroscopy.
b) The Synthesis and Characterization of $[Mo(CO)_4(piperidene)_2]$ and its Triphenylphosphine Derivatives – A study of the reactivity of a transition metal carbonyl and an introduction to the use of physical methods to obtain structural information.
c) Preparation and Properties of Ferrocene – A study of an organometallic sandwich compound and the effect of a metal on reactivity and properties of an organic ligand.

d) Preparation and Properties of Cobaloximes: Vitamin B_{12} Model Compounds – A study of an inorganic coordination complex useful as a model to illustrate the unconventional properties of Vitamin B_{12}.
e) Synthesis and Characterization of $YBa_2Cu_3O_{7-x}$ – A High Temperature Superconductor –The preparation and characterization of a high temperature superconductor, a ceramic material that conducts current with zero resistance when cooled.

III. Laboratory Safety

WARNING: Unconventional experiments can be dangerous, most chemicals are toxic, and no student (this includes you!) is immune to accidents. Every experiment in this course carries with it some element of danger; if you do not feel that you have the time, facilities, or proper level of preparation to do an experiment with caution and care, then don't do it. See the instructor or TA instead.

The Right to Know Law states that you have the right to be informed of any risks associated with your work in this laboratory. You already know of the general risks and hazards involved in working with common glassware, solvents, and chemicals. When an uncommon chemical or piece of apparatus is called for in an experiment, the laboratory write-up will point you to any extra cautions which might need to be observed with those materials. MSDS sheets detailing the physical properties and potential physiological effects of all the chemicals used in this course are on file in the University Safety Office; you may examine these at any time. In general, before you work with a chemical for the first time, it is good practice to look it up in the Merck Index or other reference manual to see what kind of extra precautions, if any, might be in order.

a) **SAFETY GOGGLES ARE TO BE WORN IN THE LABORATORY AT ALL TIMES!** No exceptions: The first infraction will result in a warning; the second will result in expulsion from the lab and a failing grade for the experiment in progress. We are serious about this!
b) No variations on the experiments as written may be performed without the explicit approval of the instructor.
c) Coming to laboratory unprepared to do the experiments carefully and cautiously is a hazard for all the students in the class, and this may lead to your dismissal from that week's session. If you don't understand the instructions or risks associated with a procedure, don't do it; see the instructor or TA instead.
d) BE CAREFUL handling chemical reagents. Avoid contact with the skin and breathing vapors. Almost any chemical can be toxic, if absorbed into the body in large enough quantities. Conversely, virtually any reagent can be manipulated safely when proper procedures are followed. Thus, follow safety procedures outlined on the container or in the preparation directions. If you have any questions, be sure to check with the instructor or TA.
e) In case of spills, inform the TA and find out the proper cleanup and disposal procedures to be followed.
f) Be sure you KNOW the location of all safety equipment (eye washes, fire extinguishers, etc.), and what to do in the event of a fire, a chemical spill or exposure, etc.

g) To reiterate: there are risks associated with all chemical experimentation. We have tried to choose experiments in which those risks are minimal; with the proper care, all the procedures in this course can be (and have been!) carried out safely and successfully. You can maximize your own safety and the safety of your classmates by being sure that you understand how to carry out a procedure, and handle chemicals, or to ask for help BEFORE you attempt any experiment.

IV. Preparation

Two weeks have been allotted for each experiment in this course; adequate preparation before coming to lab is necessary to make the most effective use of this time. The work has been planned so that each student will have to answer questions about the experiment and interpret the data obtained in the laboratory. You are urged to become familiar with the literature of inorganic chemistry and should consult it when answering the questions, but the use of reference books is not intended to be a substitute for the performance of laboratory exercises.

The experimental procedures and questions to be answered will be provided to you at the beginning of the course and should be studied thoroughly prior to starting work in the laboratory.

V. Grades

Final grades will be determined as follows:

a) Laboratory reports (67%): The reports, each of equal weight, will be due **one week** after the completion of the experiment. No lab reports will be accepted for grading after the last scheduled meeting of the lab. Late reports will be penalized 2 points for each day they are late.
b) Laboratory technique and preparation (13%): This portion of the grade will be assigned by the TA's based on preparation for and performance in the lab.
c) Laboratory final exam (20%): The final will be given during the last meeting of the lab and will cover theory and procedures used in the experiments during the semester.

VI. Lab Reports

Writing Lab Reports

Laboratory experiments are usually written in the third person. There are eight distinct sections that are crucial to the report of any published journal article:

a) Title and Author
b) Abstract
c) Introduction
d) Experimentation
e) Results
f) Discussion
g) Conclusion
h) References

Each one of these sections will be discussed in greater detail herein. In addition to providing a brief explanation, a sample scientific report displaying these components will be supplied for you to refer to.

Evaluation Guidelines:

Title

- Describes lab content concisely, adequately, appropriately

Abstract

- Conveys a sense of the full report concisely and effectively

Introduction

- Successfully establishes the context (scientific concept, lab procedure) of the lab
- Effectively presents the objectives and purpose of the lab
- Presents interesting questions or issues related to the lab

Experimentation

- Gives enough details to allow for replication of procedure

Results

- Opens with a sentence or two describing the main finding(s) of the lab
- Presents visuals clearly and accurately
- Presents verbal findings clearly and with sufficient support
- Successfully integrates verbal and visual representations

Discussion

- Opens with an explanation of how the findings link to the context of the lab
- Addresses questions and issues related to the lab and discusses answers to these questions
- Sufficiently addresses other issues pertinent to the lab

Conclusion

- Convincingly describes what has been learned by doing the lab

Helpful Books/References:

1. R. S. Drago, Physical Methods for Chemists 2nd Ed., W. B. Saunders, 1992.
2. D. F. Shriver, P. W. Atkins, and C. H. Langford, Inorganic Chemisty 3rd Ed., Freeman, 1990.
3. J. E. Huheey, E. A. Keiter, and R. L. Keiter, Inorganic Chemistry: Principles of Structure and Reactivity 4th Ed., Harper Collins, 1993.
4. G. L. Miessler, D. A. Tarr, Inorganic Chemistry 3rd Ed., Pearson Education, 1991.
5. F. A. Cotton, G. Wilkinson, C. A. Murillo, and Manfred Bochmann, Advanced Inorganic Chemistry 6th Ed., John Wiley and Sons, 1999.
6. L. Pauling, The Nature of the Chemical Bond 3rd Ed., Cornell University Press, 1960.

7. R. H. Crabtree, The Organometallic Chemistry of the Transition Metals 3rd Ed., John Wiley and Sons, 2001.
8. F. A. Cotton, G. Wilkinson, and P. L. Gans, Basic Inorganic Chemistry 3rd Ed., John Wiley and Sons, 1995.
9. www.ncsu.edu/labwrite/

VII. Lab Notebooks

The laboratory notebook must be bound, not looseleaf. Notebooks will be graded; your entries must be neat and in pen.

Before lab: Write the title and a brief introduction/purpose statement on the page. Write out all reactions and figure out molecular weights of all species involved.

During lab: Write the procedure as you do it, actual amounts used (calculate numbers of moles), all observations and results, and the date.

You need not use complete sentences, and you may use outline form if you wish. Be sure to include all information: Someone else should be able to complete the experiment using only your notebook as a guide!

VIII. References

General texts which may be helpful are:

W. L. Jolly, The Synthesis and Characterization of Inorganic Compounds, Prentice-Hall, Inc. 1970.

J. E. Huheey, Inorganic Chemistry, Harper and Row (several editions).

F. A. Cotton and G. Wilkinson, Advanced Inorganic Chemistry, Wiley Interscience, 5th edition, 1988.

K. F. Purcell and J. C. Kotz, Inorganic Chemistry, W. B. Saunders, 1977.

R. S. Drago, Physical Methods in Chemistry, W. B. Saunders, 1977.

B. Douglas, D. H. McDaniel, and J. J. Alexander, Concepts and Models of Inorganic Chemistry, Wiley, 2nd edition, 1983.

D. F. Shriver, P. W. Atkins, and C. H. Langford, Inorganic Chemistry, Freeman, 1990.

Z. Szafran, R. M. Pike and M. M. Singh, Microscale Inorganic Chemistry, Wiley, 1991.