

Chemistry 317, Winter 2003

Inorganic Chemistry Laboratory

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A section: discussion hour Tuesday 8:30 - 9:20 am in BAG 106

B section: discussion hour Tuesday 9:30-10:20 am in BAG 106

A & B sections: W 1:30 – 4:50 & F 1:00 - 4:20, Bagley 293

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Introduction to Chemistry 317

Welcome to Chemistry 317, Inorganic Chemistry Laboratory. Our goals are to make this a stimulating, challenging and useful experience. You will be introduced to new techniques and new kinds of chemicals and chemical reactivity. The class will tie together material you have had in lecture courses, and will ask you to design and improve experiments. There are some lab periods for which no instructions are provided; you must choose what you want to do and invent a procedure to do it. We will help you, but we want you to bring your insights, enthusiasms, questions, and skills to the course. Some of the material will be familiar, while other parts of the class will be quite new. Some of the experiments work like a charm, others we are still perfecting – and we hope you will help us make them better. We are eager for your suggestions and comments.

Safety

In this laboratory—as in any laboratory—there are a number of hazards. Learning how to deal with hazardous situations safely is an important part of what you will learn in the class. If chemistry majors cannot handle hazardous situations involving chemicals, then who in the society can? It's usually chemistry majors who write the rules for safe handling of chemicals. Safety is an important focus of this class and we want you to think about safety as you read this lab manual and, especially, as you work in the lab. There will likely be a safety question on the exam. There was a scary incident during a Chem317 lab period eight years ago but fortunately no one was hurt.

The most important safety rule is to THINK! Good common sense will get you through most situations. If there is anything that is unfamiliar or doesn't seem right, stop what you are doing and ask. There is a TA for every eight students in the lab, so there should always be someone nearby to assist or explain. You will be using a number of expensive pieces of equipment during the lab, so it is important that you understand how this equipment works. Don't just plow ahead if anything looks wrong. No one will be criticized for asking. It is, however, critical that you arrive prepared for the laboratory, having worked out the procedures in your own mind so you know what you're going to do.

There are a few safety rules we will strictly enforce. Safety goggles must be worn at all times in the lab. Eating in the lab is of course forbidden. Shoes must be worn at all times (no sandals or open toed shoes), and no shorts or short skirts.

An increasingly important part of safety and safe handling of chemicals is their disposal. The disposal of the solutions and products in each lab experiment is either described in the lab write-up or your TA will explain the procedures. Note that there are waste bottles for each lab, as well as for rinse solvents (acetone, ether...), acids and bases. Only chemicals go in these bottles. Syringe needles go in the sharps bin, and glass products (including pipettes) go in the glass waste. Place all waste in the appropriate container. If you aren't sure where it goes, ask your TA. Leave all "smelly" items in the hood with appropriate labels. When in doubt, put your waste in a bottle and label it to indicate the contents. No potentially hazardous waste should be disposed of down the drain or allowed to evaporate into the fume hood. [Environmental Health and Safety even views Coca-Cola™ as "potentially hazardous waste"!]

Logistics and Schedule

Chemistry 317 consists of two laboratory periods and one “lecture” hour per week. The experiments are designed for students to work in pairs, with a maximum of 16 students (8 pairs) in the laboratory. In each lab period, half of the students will work on one experiment and the other half will work on another. Those in the AA section will be doing experiments from the first column below, and must come to the Monday 8:30 a.m. discussion hour. Those in the BA section will work on the second column and must come Monday at 9:30 a.m. In this way the discussions will be related to the experiments you are doing.

The discussion hours will include some lecturing, to provide background and understanding of the experiments. But primarily these hours will be forums for discussion of the lab just completed—for instance how to analyze your spectra or numerical data. Please bring your data, your questions, and your opinions! Most students have found these sessions quite helpful. Attendance will be taken.

The schedule for the class is given below; the write-ups for each of the eight experiments make up the body of this lab manual.

Lab period #	Section AA	Section BA	date
1	Check in	Check in	Jan 7
2	Chromous Acetate	Chelate Effect	Jan 9
3	"	"	Jan 14
4	Chelate Effect	Chromous Acetate	Jan 16
5	"	"	Jan 21
6	Phosphorous Acid	ZnS phosphor	Jan 23
7	"	(Arene)Mo(CO) ₃	Jan 28
8	BF ₃ •NH ₃	Chelate Effect II	Jan 30
9	(Arene)Mo(CO) ₃	Linkage Isomers	Feb 4
10	Chelate Effect II	"	Feb 6
11	Linkage Isomers	"	Feb 11
12	"	"	Feb 13
13	"	(Arene)Mo(CO) ₃ II	Feb 18
14	"	"	Feb 20
15	(Arene)Mo(CO) ₃ II	"	Feb 25
16	"	Linkage Isomers II	Feb 26
17	"	Phosphorous Acid	Mar 4
18	Linkage Isomers II	"	Mar 6
19	ZnS phosphor	BF ₃ •NH ₃	Mar 11
20	Check out	Check out	Mar 13

Even with only four pairs of students working on a given experiment, there will occasionally be times when you will have to wait to use a piece of equipment. Try to find something else that needs to be done while you're waiting. Making efficient use of your time is a critical laboratory skill (and a skill you will be graded on). Within each experiment, you and your lab partner will often be doing different things. You should try to follow what she or he is doing, as the final lab write-up will require both of your data. But—don't worry—no one will be penalized because their lab partner didn't finish or something like that.

Readings

It is imperative that you carefully read the lab descriptions before entering the lab. Even more than reading, you must think through what you will be doing. This is critical for safe working in the lab, and to manage your time efficiently. The TAs may take various steps to insure that the reading is carefully done.

The lab descriptions contain occasional references to the "original literature," the scientific articles which originally reported the chemistry. These are given in the standard American Chemical Society (ACS) reference format: Journal Title year, volume #, page. These and other articles are available in a folder in the 317 lab, along with various reference books. The folder and books are also on reserve at the Chemistry Library. These extra readings are optional, but may be quite useful and interesting. In the arene-molybdenum-tricarbonyl experiment, for instance, many students have found these papers useful when they design their own procedures. The extra readings provide background to help you understand your observations and better interpret your data—both critical to good lab reports.

Grading/Assignments/Notebook

You must keep a good notebook in this laboratory (and in all scientific labs). Use a bound book that pages cannot be removed from. Your notebook is your diary of what you did, and it should be written as you are working. Do not make notes on scratch paper and transcribe them into your notebook. The book should include numerical data (weights, volumes, voltages, etc.), procedures (A was added to B dropwise over 20 minutes using an addition funnel), and observations (it turned green after half the A was added). The most important features of a good lab notebook are clarity and completeness. You should never remove a page or plan to go back and fill in something later. If necessary, you can cross something out or recopy something for clarity, just indicate why and make sure the original is still legible. The notebook is not a handy piece of scratch paper. It should enable you to reconstruct what you did, including good and bad aspects of the procedures. A TA will look at your lab book periodically and may collect it at some point.

Each lab write-up in this manual ends with a description of the required assignment for the lab. All assignments are due one week after completion of the experiment. Assignments must be typed, double spaced, except for tables, figures, drawings, graphs, and equations which can be done by hand.

Three of the experiments require formal lab reports, as explained in their write-ups. The most critical aspects of any lab report are *clear thinking* and *maintaining your focus on the important issues*. Grading will be based not only on the science but also on clarity and writing skills. It's hard to judge the science if the writing is poor. This is a "W class," so you will earn writing credit (with an emphasis on earn). An introduction to writing lab reports is given on the next page.

In addition to the lab write-ups, there will be a brief (50 min) exam. This is scheduled for Thursday, March 13, 2002 at 8:30 or 9:30 a.m. (your choice). This is not a scheduled course meeting time, so please reserve that date and time on your schedule now. If you have a schedule conflict, contact Professor Mayer as soon as possible.

Your grade will be based on the formal lab reports for three of the experiments, the shorter assignments for the other labs, your lab notebook, the exam, and on your overall ability in the laboratory (as judged by your TAs). The point distribution is outlined below. The score for lab skills given by the TAs is quite important. They will be looking at how prepared and punctual you are, how well you use your lab time, your lab safety, lab awareness, and overall helpfulness (especially to your lab partner), and the quality of your ideas, suggestions, and questions.

Lab Reports	
Chelate Effect	16%
Arene Molybdenum Tricarbonyl	16%
Linkage Isomers	22%
Other Experiments	
Chromous Acetate	4%
Phosphorous Acid	4%
$\text{BF}_3 \cdot \text{NH}_3$	4%
ZnS phosphor	3%
Lab Skills (TA input)	16%
Exam	15%
Thursday, March 13 at 8:30 or 9:30 a.m.	
New Chemistry Building (CHB) 102.	