CHEM 4711WAdvanced Inorganic Chemistry Laboratory **Spring 2017**

Instructor	Connie Lu, clu@umn.edu	Smith 326
Recitation	Tu, Th 11:15 am – 12:05 pm	Smith 231
Laboratory	M – Th 12:20 – 4:10 pm	Kolthoff 491

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This course is designed as a "controlled research experience" in inorganic chemistry. Four experiments are scheduled:

A. Synthesis and Characterization of Potassium Nitrosodisulfonate, $K_4[ON(SO_3)_2]_2$, Fremy's Salt
B. Organometallic Chemistry: Synthesis of Cyclopentadienyl Derivatives of Iron Pentacarbonyl
C. Synthesis and Photoelectrochemical Studies of $(\eta^5\text{-}C_5H_5)Fe(\eta^6\text{-toluene})PF_6$
D. Determination of the Configuration Equilibrium of a Four-Coordinate Nickel(II) Complex by Magnetic Susceptibility
E. Design a Nickel Catalysis Experiment

In each experiment, several compounds are synthesized, characterized, and studied using appropriate physical/spectroscopic techniques. It is expected that you will attend lecture and laboratory on all scheduled days (attendance will be checked). You will need to manage your laboratory time effectively in order to complete the experimental work. For each student, the specific sequence of experiments can vary. Hence, the lecture topics may not correspond to the specific experiment you are working on at that time. Lectures for experiments A-D are scheduled for the first six weeks of the semester. Two lectures for experiment E are scheduled in April.

Summary of important times and dates in this class

Lab work begins	Monday, January 23
Preliminary questions due	*before* you start the experiment
First draft deadline for the 1st experiment report	4:00 p.m. Friday, February 3
Deadline for the revised 1st experiment report	4:00 p.m. Friday, **February 10**
Report for 2nd experiment assigned is due	4:00 p.m. Friday, **March 3**
Spring break	March 13 to March 17
Report for 3rd experiment assigned is due	4:00 p.m. Friday, **March 31**
Report for 4th experiment assigned is due	4:00 p.m. Friday, **April 14**
Lab work ends	Thursday, April 27
Experiment E write-up is due	4:00 p.m. Monday, **May 1**
Lab checkout	May 1 to May 4
Final exam (1 hour)	2:00 p.m., Tuesday, **May 9**

Tentative Lecture Schedule

Date	**Lecture topics**
1/17, Tu	Syllabus, Safety
1/19, Th	*no class*
1/24, Tu	Electron paramagnetic resonance spectroscopy
1/26, Th	Magnetism
1/31, Tu	Magnetism, report writing
2/2, Th	Magnetism
2/7, Tu	Infrared spectroscopy
2/9, Th	Infrared spectroscopy
2/14, Tu	Photochemistry
2/16, Th	Photochemistry
2/21, Tu	*no class*
2/23, Th	Electrochemistry
2/28, Tu	Cyclic voltammetry
3/2, Th	Cyclic voltammetry
3/7, Tu	*no class, lecture break until 4/6*
4/6, Th	Catalysis
4/11, Tu	Literature paper on nickel catalysis
4/13, Th	Catalysis
5/9, Tu	**Final exam (location TBD)**

Grading: Your grade in this course is determined by the number of points you accumulate. A total of 150 points is possible. Each day that you attend lab or you attend one of the instrument tutorials, you will receive 1 point (up to a total of 20). Each experiment (A to D only) is worth a maximum of 25 points. Both the write-up for experiment E and the final exam are each worth 15 points. For the experiments, a numerical grade (between 1 and 25) and a tentative letter grade (A, B, C, D, or F) will be assigned. **Only the first report will receive feedback prior to grading (see below for more details).** Grading of the lab reports will be based on the *results* (including quality of samples and characterization data), *discussion* of results, *answers* to questions and preliminary questions, *organization*, *presentation* (including graphs, figures, and schemes), *writing style* and *neatness*. The reports must be typed, and they are to be **turned in at the TA office in Smith 324** on the due dates listed above. **Reports submitted after the due date will not be accepted, and you will receive 0 points for that experiment.** "I" grades will not be given; a student earning a D or F grade will result in no credit toward graduation. **It is not possible to receive a passing grade (C) in this course unless a report is turned in for each of the five experiments.**

An example of a past grading scale is as follows (+/- grades will be assigned within these ranges, as appropriate):

Total Points	Grade
132	A
108	B
84	C
<84	D or F

Writing feedback for the first report:

This course is a writing intensive course. You will have the opportunity to incorporate feedback prior to grading of the first report. The initial submission will be read and evaluated by a TA for organization, style and writing clarity. **The report may not be graded for content and experimental accuracy etc. at this time.** Comments will be made to help you revise the report and one of two evaluations will be assigned: **minor** revision required for a passing grade; **major** revision required for a passing grade. The evaluation of your first report and the report itself will be handed back to you and you will be allowed to revise it. The submissions of your remaining four reports are final, and no revisions of allowed these are allowed.

Laboratory Rules:

(a) SAFETY!!!! All laboratory operations will be performed in the safest manner possible. A mandatory safety lecture will be given on the first day of class. **Safety goggles must be worn at all times in the laboratory. Gloves should be worn whenever you are handing samples and chemicals.** Please be considerate and **take off your gloves** when touching door handles. Particular attention should be paid to the waste bottles that are in the lab. Experiment waste added to the wrong bottle can cause an explosion or fire!

(b) Lab work will begin on Monday, January 23, and end on Thursday, April 27. **(The week of May 1 − 4 is for check-out only!)** All students should check in during the first assigned lab period and learn about their assigned first experiment from the instructors. You will be allowed to begin lab work during the first week.

(c) In order to prevent overcrowding of facilities, you will be assigned a sequence of experiments. For example you might have ABCD or ADBC. Be certain to plan your schedule as early as possible and register for any reserved equipment as soon as you can anticipate with reasonable accuracy the periods in which you can use it.

(d) All work must be done in the labs of the course unless specifically directed otherwise by the instructor. It is not acceptable to run spectra, for example, on research instruments in other laboratories. In particular, students taking another lab course or doing senior thesis are not permitted to do work for this course in their other lab. All students must use the instruments provided for this course. Students working during their scheduled lab periods will have priority for instrument use. **Instructions for the operation of the instruments used in Chem 4711 are given in the Appendices at the end of the manual.** During the first weeks of class, tutorials on instrument operation will be offered. **Sign-up sheets will be available and attendance will be part of your grade.**

(e) **All computer files generated as data must be saved in your personal chemistry account created for this class. No sharing of data files with other classmates is allowed. These files are part of the record that goes with each report. Reports that are not consistent with data files on the server will be severely marked down. We routinely back up all the computer files and check student progress against the data in your files.** All files are stored on the chemistry server and can be accessed from the lab (Kolthoff 491) or from the Chemistry Microcomputer Facility (Smith 101D). Your initial account logon username is your UofM student internet ID (i.e. smith001). Your initial account logon password is your UofM student ID number. All data files collected

must be stored in your account and these accounts will be periodically checked by the TAs for authenticity. You must also save any other work such as lab reports, graphs, or other data workup files, for this course only, in your Chem 4711 account for this class. **All files generated using computers in 491 Kolthoff must be saved in your personal account, not on the local hard drive**. Files left on the local hard drives will periodically be erased, just like the policy of the Microcomputer Facility. Please read Appendix V for the detailed instructions for logging on and saving data files.

(f) **Your lab notebook must be signed by a TA every day during the laboratory period. Either a carbon copy or a photocopy of your lab notebook must be turned in with your lab report for a given experiment.**

(g) It is a policy of this course that you may not work on an experiment until you have handed in the preliminary questions for that experiment. You will be asked to leave the laboratory until the preliminary questions are handed in. You are also not allowed to work on more than two experiments at once. You may proceed to a third experiment only when the lab report for the first has been submitted. *There are no exceptions to this rule.* TAs will not permit the student to be in the laboratory until the required experiment is handed in. A lab missed for this reason cannot be made up.

(h) Lab checkout will be **May 1 − 4**.

(i) Any student found performing unauthorized experiments or behaving in an unsafe manner in the laboratory may be removed from the laboratory at any time. Whether or not behavior is unsafe is at the discretion of the instructors, and this includes failure to properly respond to instructions in a timely manner. Removal from the laboratory may be for a period of time as short as the remainder of the current lab period or as long as the remainder of the course itself, depending on circumstances.

(j) According to the CLA Classroom Grading and Examination Procedures, scholastic dishonesty is defined as: *"Any act by a student which misrepresents the student's own academic work or that compromises the academic work of another. Scholastic dishonesty includes (but is not necessarily limited to) cheating on assignments or examinations; plagiarizing, i.e. misrepresenting as one's own work any work done by another; submitting the same paper, or substantially similar papers, to meet the requirements of more than one course without the approval and consent of all instructors concerned; depriving another of necessary course materials; or sabotaging another's work."* It is your responsibility to ensure that any document you submit (data, lab reports, analysis, etc.) represents your own work. Academic dishonesty in any portion of the academic work for a course shall be grounds for awarding a grade of F or N for the entire course.

Laboratory Notebook

1. The lab notebook is to be kept in duplicate. Special notebooks designed for this with alternate white and yellow pages are available in the bookstore and must be used. Since the grading will be done on the carbon copy, all entries must be made with ball-point pen so that the carbon is clearly legible.

2. Each page of the notebook should be headed with your name; the date; the title: Preliminary, Data, or Write-up; the experiment number (only one experiment should be included on any given page): and in the case of Data sheets, the instructor's/TA's OK. It is a good idea to reserve a section of your notebook (10-15 pages for each experiment).

3. Preliminary Questions (PQs) - In each experiment there is a set of questions to be answered before the experiment is begun. These are to be answered in the lab notebook, and you should hand in a copy of these questions before you start the lab work for the experiment. **The preliminary questions that you hand in prior to beginning an experiment are graded and count toward the 25 points for each experiment.** The preliminary questions for each experiment should be handed in on separate sheets (i.e. do not mix PQs for Experiment A on the same page with those of Experiment B). In some of the questions you are asked to make calculations about quantities of material to be used in a synthesis. If you feel there are other preliminary calculations that should be made before you come to lab, these should be included with the preliminary questions. The TAs will keep track of whether you have turned in the PQs for the experiment that you are working on. If you have not handed in the required PQs, you will be asked to finish and turn them in before you may proceed with your lab work.

4. Data - All data collected and experimental observations made during the course of the experiment are to be recorded on the Data sheets. You are not asked to copy the experimental instructions although you may if this is the way you prefer to keep a lab notebook (this is a point on which good research workers differ). You are required to record all numerical data. For example, if the instructions say to put 25 ml of 6 M HCl in a 250 ml beaker, you should record the volume and concentration in your notebook as you do it; the beaker size may be omitted. If you record weights, the weights of the empty container, of the container plus sample, and of the difference should all appear if they were necessary to measure the weight. In reactions, color changes, precipitates, and evolved gases should all be noted. The temperature should be recorded if there is any possibility it will matter later. All of this information should be recorded directly into the lab notebook. Near the end of the period (before 4:00 p.m.) the TA should sign your data notebook. When you hand in the report for an experiment, you should also hand in either a carbon copy of your data or a photocopy.

   In past offerings, students have worked in pairs on experiments. Students are expected to show up to lab on a consistent basis. **If you are not present when data is collected by your partner, then that data will not be made available to you.** TAs will monitor attendance to ensure that both partners are making equal contributions.

   (Note: Writing lab reports should be done individually. All reports are checked for plagiarism against the internet and against lab reports in the class.)

Laboratory Reports

Your report must be typed. A hard copy of your report will be turned in and the computer file should be saved in your work file. This means that your entire report must be contained in the computer file. A report should have a title, an *introduction*, an *experimental*, *results*, and *discussion* sections. For more guidelines, see Appendix VI. **The graders already have copies of the lab manual, so there is no need (in fact, it is very negative!) to reprise the writing in the lab manual as part of your report.** The preliminary questions that were handed in before you started the experiment need not be re answered in the report because they are assigned points based on your previous answers. You must include all tables of data, graphs, charts, spectra, etc. in the *results* section.

If you have questions concerning the specific content for an experiment report, ask the TAs.

The *experimental* section can simply say that the procedures given in the lab manual were followed exactly but you should mention any deviations or unusual observations. The *results* section should include the experimental data and calculated values which should be displayed in tabular form; graphs are always appreciated when appropriate. A graph always has (1) a caption/title (2) and labels on the axes (appropriate variables and units). An analysis of error is required (i.e., a number is not worth reporting without an error estimate). The necessary calculations should all have been made in the lab notebook on Write-up pages. Details of required calculations are given in each experiment. The method of calculation should be shown and described in every case as well as the intermediate steps in the calculations. If graphs are necessary, they should be made in Excel. All spectra and samples should be turned in with the report. Be sure to describe what you did in words for each experiment. Do not copy the procedure in the hand out, but describe how you worked up your data and did the calculations. Include key equations and formulas and show intermediate results in tables. Assume that you are writing this for someone unfamiliar with the experiment. Also, assume that you are writing this for your boss and your salary depends on a good job!

The *discussion* section should include a general discussion of the results. Answer the required questions here. At the end, include a section entitled *suggestions for improving this experiment*. You should critique the experiment in this section. This is helpful to us.

Turning in Samples

After you have spent a number of hours preparing a sample you should spend a few additional minutes putting it in a suitable container before turning it in. The following suggestions should be followed.

1. Choose a container large enough for the sample. Do not turn in several small bottles unless there are significant chemical or physical differences in the samples.

2. Unless the sample is light sensitive, it should be in a colorless bottle so that the color and condition of the sample may be seen.

If the sample is light sensitive, use an amber bottle, or in the extreme case cover the bottle with paper.

3. Consider whether the sample is sensitive to the atmosphere. A slightly sensitive substance should be placed in an ordinary bottle that is then sealed with paraffin.

4. Consider whether the substance is reactive. Very often a cork or rubber stopper will be attacked by the compound, in which case a glass stopper should be used.

5. The bottle should have substantial walls and stand upon the shelf. Test tubes, flasks, and weighing bottles are not acceptable containers. A large fraction of the time a plastic-capped vial is adequate.

6. The sample should be well labeled. The name and formula of the compound, the name of the maker, and the date of the preparation should all be clearly indicated.

Some "Last Words" before you begin:

In this course you should keep three aims in mind. You should broaden your knowledge and understanding of inorganic compounds and their reactions (read the references!), you should learn to apply certain basic preparative techniques, and you should learn to apply certain physical measurement techniques to the study of your compounds.

It is important to use your time efficiently in this course. Many of the experiments, particularly the preparations, have long periods in the course of the experiment in which you are just waiting. Plan to work on other experiments, collect necessary characterization data, or work on your reports during this time. **Remember that the quality of your characterization data will be graded**. You may need to collect multiple spectra (and vary conditions) to obtain a decent one. Also keep in mind that not all experiments go exactly as planned the first time you attempt them, and you may need to repeat synthetic or characterization steps as necessary.