Chemistry 5530 Laboratory- Part I
Spring Semester, 2008

Professors in Charge: Part I: John L. Hubbard, ML 361, 797-1641, jlhub@cc.usu.edu
Office hours- MW 12:00 and by appointment
(Part II: B. Davidson - Separate Documents to be provided)

In accordance with the Americans with Disabilities Act, reasonable accommodation will be made for all persons with disabilities in order to assure equal participation in Chem 5530. Please meet with Dr. Hubbard during the first week of labs to make arrangements.

Meetings: Monday and Wednesday- 1:30-5:20 pm is the official time slot. Due to the nature of the experiments, the lab rooms ML 374 and ML 370 may be open at other times, as necessary.

Lab Fee (required): $55.00 used for the purchase of equipment and supplies for the lab.

Lab Text : Lab procedures are provided via the internet WebCT Site

Lab notebook : Data must be recorded in a bound notebook. I will check this periodically for content, organization, neatness, credit.

Course Objectives: Chem 5530 Lab (Part I) has laboratory experiments designed to cover a range of advanced techniques used for the synthesis of inorganic and organometallic compounds. Students will learn how to work on a vacuum line, perform Schlenk line mainipulations, use photochemical apparatus to prepare air-sensitive solvento complexes, and use a variety of highly reactive chemicals. The some experiments involve muliti-step syntheses of considerable complexity. A total of 5 syntheses are planned for Part I of the course and will constitute one half of your Chem 5530 grade.

Pre-Lab: 1 page – submitted by **BlackBoard** (E-mail to JLH). Due before you begin work. **(10 pts total for each lab)**

1. Describe the location of the chemicals and equipment needed for the procedure.
2. Briefly summarize the steps of the reaction(s) with chemical reactions
3. State any special precautions necessary.

Lab Write-up: Type-written reports **(90 pts total for each report)**

1. Introduction describing the experiment, type of reactions, basic type of product(s) to be formed and isolated. **(10 pts)**
2. A brief description of the procedure should be given. Include reaction equation(s), reactant and product quantities in grams and equivalents (millimoles), and yields as percentages. Sketch the basic experimental set up (can be done by hand if neat). Include Labeled spectra (or copies) **(20pts)**
3. Discussion: Note special features of the procedure (air-sensitive steps, etc), briefly discuss how your results met expectations (yields, purity, etc), the basic *"philosophy"* of the reaction, what bonds are broken, what bonds are formed, what intermediates are (may be) involved. Discuss the energetics (exothermic, endothermic) and the effects of applied heating, cooling, pressure. Interpret the spectroscopic data, using carefully labeled "processed" spectra (don't just include raw data!) **(30 pts)**
4. Summary: Why is the product potentially interesting, what features of the synthesis that will likely be the most memorable? **(20 pts)**
5. Overall neatness, clarity, grammar, punctuation, etc. **(10 pts)**

5 prelabs @ 10 pts =50 pts 5 reports @ 90 pts = 450 pts
Total points = 500 pts

Grading: 100-90% A 80-89% B 70-79% C 60-69% C

Part 1 Experiments: **Work in pairs**

Experiments:

1. Preparation of Germane (GeH_4) on a vacuum line manifold
2. Photochemical Ligand substitution on $(\eta^5\text{-}C_5H_4CH_3)Mn(CO)_3$ – Synthesis /NMR of an η^2- styrene complex
3. Synthesis and characterization of Nanoparticles
4. Synthesis and characterization of polymers
5. Synthesis and characterization of $[\eta\text{-}(C_7H_8)Mo(CO)_3]$ and $[(\eta\text{-}C_7H_7)Mo(CO)_3]BF_4$

Schedule- Part I ***15 lab days***

	Date	***Experiment "cycle"***
January	Wed 9,	Orientation-
	Mon 14	
	Wed 16	
	Mon 21	-Holiday
	Wed 23	
	Mon 28	
	Wed 30	
February	Mon 4	
	Wed 6	
	Mon 11	
	Wed 13	
	Tues 19	(a Monday Class day)
	Wed 20	
	Mon 25	
	Wed 27	finish up loose ends

****

Part II- begins March 5 (Dr. Davidson)

Advanced Synthesis Laboratory Chem 5530
Learning Objectives

Assemble glassware together with mechanical accessories (e.g. heating mantels, stirring equipment, vacuum pumps, cooling sources, U.V. lamps) to perform synthetic chemical procedures.

Safely handle cryogenic materials like liquid nitrogen and dry ice.

Safely handle toxic and highly reactive chemicals, active metals, compressed gases.

Utilize Schlenk and high-vacuum manifolds to generate and manipulate volatile substances and perform chemical reactions under an inert atmosphere.

Perform vacuum distillations and trap-to-trap purification sequences using frozen slushes prepared from liquid nitrogen and organic liquids.

Design reactions so that intermediate sampling can be performed to monitor the reaction progress by spectroscopy.

Prepare samples for IR and NMR spectroscopy, obtain data, interpret spectra.

Prepare liquid chromatography columns under inert atmosphere and collect fractions from the column under inert atmosphere.

Set up reactions using pressurized gaseous reagents in lecture bottles and larger cylinders.

Experiment with techniques of recrystallization (vapor diffusion, solvent layering, solvent mixtures).

Learn to work with chemicals inside an inert atmosphere dry box.

Write concise reports of synthetic procedures with interpretation of results and spectral data.