Syllabus for
CHM 4915
Advanced Laboratory
Spring 2010

Course Description: An interdisciplinary laboratory course featuring experiments in inorganic, organic, and organometallic chemistry. Experiments will emphasize advanced techniques of chemical research, including the synthesis and characterization of important classes of inorganic, organic, and organometallic compounds. This course is designated a writing-intensive course.
Prerequisites: CHM 2845, CHM 4900.

Course Goals: Students will:

(1) Identify and describe information pertaining to substances, reactions, characterization methods or other specialized methods needed for each experiment from various chemistry resources.
(2) Collect and record data and apply techniques relevant to experiment objectives.
(3) Describe chemical principles that underlie each experiment.
(4) Apply physical methods of analysis to determine structural, electronic, magnetic or other properties of substances.
(5) Analyze (collected) data for the purpose of characterizing substances, testing hypotheses, or interpreting experimental results.

Meeting Times: 12:00 P.M. – 12:50 P.M. Monday (lecture)
2:00 P.M. - 4:50 P.M Tuesday, Thursday (laboratory)
Meeting Place: Physical Science Building 4125 (lecture), Physical Science Building 4010 (laboratory)

Instructors: Dr. Mark E. McGuire, 3430 PSB, 581-6228, memcguire@eiu.edu
Office Hours: Mon. 3:00-3:50 PM; Thur 9:30-11:30 AM; Fri 9:00-9:50 AM
Dr. Ed M. Treadwell, 4450 PSB, 581-6229, emtreadwell@eiu.edu
Office Hours: Mon. 1:00-2:00 PM; Thur noon-1:00 PM; Fri noon-1:00 PM

Texts: (1) *Synthesis and Technique in Inorganic Chemistry*, 3rd ed, Girolami, Rauchfuss, and Angelici
(2) *Inorganic Chemisry*, 3rd ed., Miessler and Tarr
(3) *Advanced Organic Chemistry*, 6th ed, March
(4) *Introduction to Spectroscopy*, 3rd ed, Pavia, Lampman, and Kriz (PLK)
(5) *Organic Chemistry*, 6th ed, Wade
(6) *Organic Laboratory Techniques* by Pavia, Kriz, Lampman, Engel

Format: This class will meet most weeks on Monday for pre-/post-lab discussions and exams, while Tuesday and Thursday meetings will be used to conduct the experiments and collect experimental data. A list of experiments and a tentative timeline is given later on in the syllabus. After each experiment is concluded, a written report will be required, according to the guidelines in the syllabus. There are three components to the course grade:

- lab notebook, 15%
- lab reports, 65%
- tests (mid-term and final), 25%

A standard grading scale (90% + = A, 80-89% = B, etc) will be employed.

Course Policies:

1. **SAFETY IS VERY IMPORTANT. You must be wearing your university-approved lab goggles at all times, as well as working carefully according to the directions given and aware of the hazards involved in each experiment. Sandals, open-toed shoes, and shorts are prohibited.**
2. Please inform us of any preexisting medical conditions you have as soon as possible, or if you have a documented disability and wish to discuss academic accommodations.
3. If you are absent because you were sick, you will be allowed to make up the lab/quiz if you provide an excuse that is *written* and *signed* by a medical official. If you must be absent for a lab period because of required travel with an athletic team, please arrange accommodations BEFORE you leave. Absences for emergency reasons will be judged on a case-by-case basis. Lack of a valid reason for an absence will result in a zero score on the quiz that was missed. If the first day of multiple-day experiment is missed without an acceptable excuse, you will NOT be allowed to perform the lab and will receive a zero on the lab report.
4. Beginning at 2 P.M. on the day they are due, reports turned in after the deadline will incur a late penalty. For the first late report, the penalty will be 10% for the first day and 25% each day thereafter (including weekend days). Any subsequent late reports will be assessed a 25% penalty per day, starting on the first day. More than 3 late reports will result in the automatic lowering of your course grade by 1 letter grade.
5. EIU's policy on academic integrity (as described in the EIU Undergraduate Catalog and Student Conduct Code) applies to all parts of the course. Copying background information, results, answers, and calculations from someone else's lab report, creating or falsifying data, or plagiarizing from the course materials/literature, constitutes cheating and is grounds for an **"F"** in the course. The program TurnItIn will be used to monitor for plagiarism, and you must sign up for this course. The class id is **3036125** course password is "**chm491510**". Late postings to TurnItIn count the same as handing in a late report.
6. A carbon-copy laboratory notebook with at least 50 pages will be required for this class. Record all information in the laboratory notebook using ink only, in legible handwriting. Copies of your lab notebook pages must be turned in at the end of each lab period.
7. A $10 fee will be billed to your University account. This fee covers the cost of consumables (*e.g.* chemicals, glass and rubber tubing, filter paper, etc.). It also covers the cost of items, except those in the glassware kit, accidentally broken while performing scheduled experiments. You will be assessed a breakage fee (equivalent to replacement cost) for any item(s) in your glassware kit broken during the course of the semester or missing from it at checkout.

Tentative Experiment and Report Schedule[1]

Week	Date	Experiment	Report Due
1	Jan 12	Check-in and orientation	
	14	Exp 1: Catalytic Hydrogenation	
2	19	Exp 1: Product Analysis	
	21	Exp 1: Product Analysis	
3	26	Exp 2: Synthesis and Characterization Vanadium Phosphate	
	28	Exp 2: Product Analysis	Exp 1
4	Feb 2	Exp 3: Kinetic vs. Thermodynamic Control in Enolate Formation	
	4	Exp 3: Product Analysis	
5	9	Exp 3: Product Analysis	Exp 2
	11	Exp 4: Synthesis and Characterization a Paramagnetic Coordination Compound	
6	16	Exp 4: continued	
	18	Exp 5: Synthesis, Characterization, and Application of a Chiral Homogeneous Epoxidation Catalyst 5A: Resolution of 1,2-Cyclohexanediamine	Exp 3
7	23	5B: Synthesis and Characterization of a Chiral Ligand	
	25	5B: continued	Exp 4
8	March 2	5C: Metallation of the Chiral Ligand—the Catalyst	
	4	5D: Epoxidation using the prepared Catalyst	
9	9	5D: Column chromatography	
	11	5D: Product analysis	
10	16 18	Spring Break Spring Break	
11	23	Exp 6: Synthesis and Characterization of *trans*-$Fe(PPh_3)_2(CO)_3$	
	25	Exp 7: Isolation of Intermediate	Exp 5
12	30	Exp 7: Product analysis	
	April 1	Exp 7: Synthesis and Employment of an Evans auxillary	
13	6	6A: Formation of Amino Alcohol / Monoprotection of Glycol	Exp 6
	8	6B: Formation of Oxazolidinone / Oxidation of Glycol	
14	13	6C: Acylation of Oxazolidinone	
	15	6D: Aldol reaction	
15	20	6E: Removal of Auxillary	
	22	Exp 8: Transition Metal Mediated Coupling Reaction	
16	27	Exp 8: continued	
	29	Check-out	Exp 7
17	3*		Exp 8

[1.] Experiments 2 and 4 are in *Synthesis and Technique in Inorganic Chemistry*. All other experiments will be distributed as handouts.

MAINTAINING A LABORATORY NOTEBOOK

Keeping a detailed, coherent laboratory notebook is an essential part of scientific research. Not only is the notebook important as a permanent record of your research, but the notebook can be essential in understanding low yields and unexpected results. A good lab notebook should allow anyone, using only your notebook and whatever references contained therein, to repeat EXACTLY your experiment and obtain the same overall results as you obtained. It is therefore extremely important that the notebook be complete AND completed as the experiment is carried out. The experimental procedure section should NEVER be filled out after you leave the lab.

The Notebook:

- Your lab notebook should be dedicated exclusively to this course, and be a bound notebook with detachable carbon-copy pages.
- All entries should be made in nonerasable ink, and should be legible. Any mistakes should be crossed out with a single line, not by scratching out completely the information or by covering with white-out.
- The front cover should contain your name and the course number.
- The first page should be used for a table of contents, with each experiment listed by title and correlated to the appropriate pages in the notebook. The table of contents should be continually updated throughout the semester, not filled in at the end of the semester.

Experiment Entries:

Each experiment should start on a separate page, and each page dated.

Before the lab for a particular experiment starts, the following items should already be entered:

- A – the title of the experiment
- B – the reaction drawn out using structural formulas
- C – a table of reagents. There should be column headings for each reagent, the amount used, the molecular weight, the moles used, and the equivalents used. Only the chemical names in the reagent column should be completed prior to the experiment (since the amounts you use may not be exactly the amounts specified in the manual).
- D – the reference for the experimental procedure

As the experiment is carried out, the steps taken should be entered in complete sentences in the lab notebook. Be sure to include the amounts used, the order of addition, times, temperatures, and **OBSERVATIONS** as appropriate. Remember that I should be able to repeat your experiment exactly from your notebook. You do not need to describe the steps in standard procedures (such as how to use a separatory funnel or the steps in a recrystallization, etc), but should draw any unusual reaction set-up. Any characterization of the product (mp, IR, GC, NMR, etc) should be mentioned, though the analysis of the characterization need not be included. If you are only taking a melting point/spectrum of your product on a different day than the rest of the experiment, the entry can be on the same page as the rest of the experiment, but with the new date written by the information.

CHM 4915 LAB REPORTS

PART A: Preparation and Submission

All reports must be prepared with word processing software. **Reports should resemble papers seen in scientific journals.**

You will hand-in one hard copy of the report and upload an electronic copy of the text portion of the report to WebCT. Both the hard copy and electronic copy are due on the date specified on the syllabus.

It is expected that your reports will be your own individual work, with your own phrasing and thoughts. You are encouraged to discuss the experiment with your peers in the class, but should be writing your own report.

Hard Copy Format

- Font: 11 or 12 point Times New Roman or Arial
- Spacing: set to 20 point or 1.5
- Margins: 1 inch on all sides
- Number pages beginning with the main body
- Page limit: 10 pages maximum excluding attachments and cover page
- Correct use of symbols, subscripts, and superscripts is expected
- Structures must be drawn using drawing software, and should be professional in appearance (reasonable bond lengths/angles, font size reasonable for structure size, etc)
- Avoid splitting tables/figures/etc across two pages, or starting a new paragraph at the bottom line of a page

PART B: Content, Format, and Style

All reports will include a cover page, the main body, references, and attachments appropriate to the report. The experiment title, date, and your name go on the cover page. **The name should not appear on any other page than the cover page.**

Divide the main body into the following sections: 1) Abstract; 2) Introduction; 3) Experimental; 4) Results and Discussion. The abstract should be on it's own separate page from the rest of the main body. Details about what to include in each section as well as what attachments to include are described below.

Abstract:

The abstract is a brief (typically < 250 words) summary of your work. It should serve to tell the reader, in summary, what you set out to do/find out, what the overall results were, and how you knew these results (key spectral characterization). **There should be some numbers / data in the abstract to lend support to your observations.**

Introduction:

The introduction is the place to discuss background material relevant to the experiment. There is no single right form for an introduction to take. Some suggestions:

- No more than 1000 words.
- This section presents the general scope of the reaction or process investigated. You should consider including a general illustrative example.
- It is often helpful to begin your introduction with a broad or overarching statement about the main idea on which the experiment is based. You should assume your reader has some background in organic/inorganic chemistry, but is not intimately familiar with the type of chemistry investigated in your experiment.
- Describe the nature of the investigation, the outcome, and the methods used to establish the outcome (in other words, what are you exploring, what are the possible outcomes, and what tools/techniques are you going to use to tell what the outcome was?). Use both words and one or more equations to describe the possible outcome**(s). Note that you should NOT talk about your results in the introduction, only what the expected results could be**.

Experimental: Divide into two sections: 1) Methods and Materials; 2) Procedure.

1) Methods and Materials:

Identify the materials, their source, and whether or not they were purified prior to use. For our purposes, Chemistry stockroom is the source of materials in containers without an identifying supplier on the label. Likewise, identify and describe specialized equipment or techniques that were used. **It is not necessary to describe common laboratory operations, such as the use of a rotovap, distillation, using a drying agent, etc..** Identify all instruments**,** pertinent operating conditions for the instruments, and sample preparation.

2) Procedure:

Divide the procedure by each compound prepared, and start each step in a separate paragraph that starts with either the name of the product or a brief description of the type of reaction being done on the starting material (include the name of the starting material).

Describe the details of each procedural step as well as significant observations and/or cues. Include amounts of materials used. In most cases, report both the mass (or volume) and moles of all reactants and isolated products. If a reactant is part of a solution, include its concentration.

Format as**:**

www g (xxx mol, xxx equiv.) substance or yyy mL (zzz mol, aaa equiv.) substance.

For solvents, it is only necessary to report the volume used. Describe all products (physical state, color, etc). Report actual and percent yields.

Be sure to report values to their correct number of significant figures.

Following the description of each procedure, refer to the compound by it's chemical name (in bold or italics), and then report the product properties that were determined: melting point,

a list of characteristic IR absorptions **in decreasing wavenumbers** (*e.g.* 3400 cm^{-1} (br)), and a list of NMR absorptions formatted as chemical shift, number of protons, splitting pattern including coupling constant(s) (*e.g.* 2.1 ppm, 2H, d, J = 2.8 Hz). Use the following abbreviations for splitting patterns: s for a singlet; d for a doublet; t for a triplet, q for a quartet; m for complex multiplet; dd for doublet of doublets; br for broad peaks, etc.. The absorptions should be arranged by either increasing or decreasing chemical shift.

For GC/MS data, report the retention time of the peak, as well as the significant / prominent ions and their relative abundance in parantheses (*e.g.* 2-bromoethane (2.45 min): 110 (54), 108 (56), 29 (100)) .

Results and Discussion: This section provides you the opportunity to report your results and describe their meaning.

To discuss the results adequately, you must clearly understand their significance. This requires that you have mastery of the theory pertaining to the experiment, and show this by mentioning the theory in your text. Your discussion must clearly point out the significance and meaning of laboratory data and observations. Clearly state any significant conclusions and either prove or properly qualify them. But be sure to **discuss** the results - give reasons for them and evidence to support them; do not merely restate them. You should comment on any peculiarities or concerns you might have about the data, as well as possible causes. When discussing product identity discuss not only the positive evidence that supports your product structure, but also negative evidence that disproves your product being simply unreacted starting material or a different possible isomeric product. Note that you do not need to repeat every peak in a spectrum, but only point out the key peaks that differentiate the starting material(s) from product(s).

Be sure to refer to figures/tables/spectra in the text proper at the appropriate place, and to give titles and numbers to all figures and tables. Refer to specific NMR peaks according to the assignments shown on the spectrum (see Attachments section).

Include footnotes to reference sources for all comparative physical data (*e.g.* melting point, specific rotation, etc.).

PART C: References and Attachments

References:

Include any and all sources consulted for this information, including those containing the procedure followed in the experiment. List by number in order of appearance in the report.

Format all references as shown:

- Journal: Author, A. B.; Author, C. D. *J. Abbrev.* **19XX,** *vol,* xx-yy.
- Book without editors: Author, A. B.; Author, C. D. *Book Title;* Series Name and number; Publisher: City, STATE (2 letters), year; Vol. 1, pp xx-yy.
- Book with editors, no authors named: *Book Title;* Editor, A. B.; Editor, C. D., Eds.; Series Name and number; Publisher: City, STATE (2 letters), year; Vol. 2, pp xx-yy.
- Book with editors and authors named: Author, A. B. In *Book Title;* Editor, C. D.; Ed.; Series Name and number; Publisher: City, STATE (2 letters), year; Vol. 3, pp xx-yy.

Attachments:

Include all that apply:

- Calculation sheet that includes all calculations except for the determination of percent yield. (Neatly handwritten in pen or pencil.)
- NMR and IR spectra. Label **the** important peaks on each spectrum. In addition, include the following annotations: compound name, line structure, and a figure number. Number spectra in the order they are discussed.
- On the NMR spectra label assigned sets of hydrogens on both the spectrum and the structure as H_a for the most downfield assignment, H_b for the next most downfield and so on.
- GC-MS: TIC and MS obtained from peaks in TIC.
- UV-vis spectra, cyclic voltammagrams, powder diffraction patterns, etc.