Advanced Synthesis Lab Chemistry 329 Fall 2018

The Instructor (contents)

- Name: Dr. Robert Badger (students usually call me Dr. Badger or Dr. B; colleagues or fellow basketball players call me Bob)
- Office: 449 Chemistry Biology Building
- Phone: 715-346-3700
- email: rbadger@uwsp.edu

Materials (contents)

We shall be using electronic notebooks for record keeping during the lab using an Apple iPad to access your electronic notebook. If you wish to access your notebook outside of lab hours, you will need your own iPad or computer(Windows or Macintosh) and will have to install the free Microsoft OneNote App.

Format:

Chemistry 329 is a lab only course that is intended to develop your synthetic skills in both organic and inorganic synthesis. We shall meet briefly at the beginning of lab on Tuesday in B130 where you will be called upon to describe where you stand with each synthetic project and what your goals will be for the days work. I will also take that time to introduce/demonstrate techniques you may not have used before. Due dates and content expectations for lab reports will be discussed during this as well.

Grading

Materials tables and balanced chemical reactions must be in your electronic notebook before work on a project may begin. This portion of each project will be worth 5 points. Daily experimental procedures must be properly entered (third person past tense in properly structured sentences) in your notebook and properly dated on the day they are performed. You must insert spectra, chromatograms, etc. into your electronic notebook with each project. The lab notebook will be evaluated on five occasions (10 points each) at random times during the semester. Reports will be submitted as word documents via the web. All project reports/samples will be worth 30 points. The samples will be graded 8 points yield, 7 points purity, and 15 points report. Quizzes (if any) will be worth 10 points each and the number given will depend on my overall evaluation of class performance in the laboratory. If laboratory performance is excellent, there may be no quizzes.

Your quiz/sample grades will also be affected by the following:

1. Ability to prepare efficiently for lab and start working promptly.
2. Ability to use materials, chemicals, and equipment efficiently.
3. Ability to assist other students.
4. Ability to execute not only written but verbal directions as well.
5. Ability to budget your laboratory time and use it effectively.
6. Ability to clean up your working area and the general lab area assigned to you.

Five points per week late (1 point per day) will be deducted from the grade for late papers, samples or quizzes. It is assumed that only class data we have agreed to share will be common to the reports you write. All other work must be yours alone, including plots, calculations and text.

There will be a final poster presentation during exam week(Dec. 17, 2018 at 10:15 AM in CBB 426) that will be worth 30 points based on your Literature Synthesis Project.

Attendance (contents)

The first hour of the Tuesday class meeting of each week will be reserved for a quiz and/or lecture to be held in CBB 426 depending on class performance and need. Attendance at all laboratory sessions is strongly recommended but will not be taken into consideration in assigning final grades. Discussion and demonstration of important theory and technique that will be used in ensuing experiments will be presented. In addition, laboratory work is very time consuming and every missed period will require three hours of later work from the students' own time. Occasional missed classes and a maximum of two hours per week of conflicts with other course times will be tolerated and may be made up outside of scheduled laboratory periods. Course conflicts should be reported to the instructor at the beginning of the semester.

In order to work outside normal lab hours,

1. the instructor must be present on the first floor of Science,
2. permission must be obtained from him before beginning work,
3. at least one other person must be present in the laboratory with you,
4. notify the instructor at the conclusion of experimentation.

No work will be allowed after 4:45 PM (a grade penalty may be imposed). A student with a very poor attendance record will not be given unlimited time to make up his/her laboratory work.

Experience has shown that many students start slowly and later find themselves short of time. Generally, this is a result of incomplete preparation and poor scheduling of time. It would be wise to read all five experiments and supplementary material carefully as soon as possible. We will begin with project 1, but you will quickly discover that a 48 hour reflux (or a seven day reflux) leaves you with time to work on other projects. Indeed, if you do not, you will not be able to complete all of the projects. Outline a detailed schedule for your time in the lab on a given project. If, during the course of your preparation, you find something you don't understand, please stop by my office. Discussing chemical synthesis and analysis with you folks is one of the most enjoyable parts of this job.

Tentative Reactions, Compounds and Projects Chemistry 329 Fall 2018

Title	Comments
Preparation a squaraine dye with unusual aromatic properties.	We shall be starting with aniline and squaric acid. Three lab periods.
Preparation and Use of a Manganese Epoxidation Catalyst	We shall resolve an enantiomer and attempt to prepare an optically active oxidation catalyst and oxidize an alkene. Six lab periods.
Prepare DMSO-d_6 and methyl iodide-d_3	We shall prepare a salt that readily undergoes deuterium exchange and determine percent deuterium incorporation via GC/MS. A one week continuous reflux.
Preparation of Nickel(II) Complexes	Taken from J.Chem. Ed. , April 1991. IR and magnetic susceptibility will be used to determine electronic structure. Four lab periods.
DEET	Preparation of insect repellant from familiar compounds. We may also prepare the dimethylamide analog to be used in physical chemistry laboratory. Three or four lab periods.
Literature Project and proposal preparation.	We will attempt to implement some of the experiments you discovered in the Literature. Six lab periods.

References

The following references are recommended as good sources for laboratory procedures, theoretical discussions, and synthetic preparations. I have also attempted to assemble a library of lab manuals in B130 for your use. Please do not remove them from the lab except to photocopy relevant pages. Please return books in a timely fashion, as everyone will want access to them at some point during the semester.

B. S. Furniss, A. J. Hannaford, V. Rogers, P. W. G. Smith and A. R. Tatchell, "Vogel's - Textbook of Practical Organic Chemistry," 4th ed., Longman, NY, 1978.(Textbook Rental)

J. March, "Advanced Organic Chemistry," 3rd ed., Wiley, NY, 1985.(QD251.2.M37 1985)

R. Adams, J. R. Johnson, and C. F. Wilcox, Jr., "Laboratory Experiments in Organic Chemistry," 7 th ed., Macmillan, NY, 1979 (QD261.A2 1979)

J. W. Zubrick, "The Organic Chem Lab Survival Manual: A Student's Guide to Techniques," Wiley, NY, 1984.(Badger's copy)

H. O. House, "Modern Synthetic Reactions," 2nd ed., Benjamin, Reading, MA, 1972.(Badger's copy)

R. T. Morrison and R. N. Boyd, "Organic Chemistry," 3rd ed., Allyn and Bacon, Boston, MA, 1973.(Badger's copy)

H. D. Durst and G. W. Gokel, "Experimental Organic Chemistry," 2nd ed., McGraw-Hill, NY, 1987.(Badger's copy)

L. F. Fieser and M. Fieser, "Reagents for Organic Synthesis," Wiley, NY 1986. Twelve volumes.(QD262.F5)

G. Saucy, Ed., "Organic Syntheses," Wiley, NY, 1985. Tested syntheses - many volumes.(QD262.07)

Z. Szafran, R. M. Pike, and M. M. Singh, "Microscale Inorganic Chemistry," John wiley and sons, Inc., New York, 1991. (QD155.S96)

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