Chemistry 380 Syllabus January 2004 Hanson

Required Texts

Silverstein and Webster, *Spectrometric Identification of Organic Compounds, 6th Ed. (1998)*

Laboratory Manual for Chemistry 380: Safety and Tricks of the Trade

Additional Resources

Your organic chemistry text will be valuable both in terms of chemistry and spectroscopy. If you don't have it available, please let me know so I can loan you one.

http://www.stolaf.edu/people/hansonr/chem380 This is our course home page.

http://www.stolaf.edu/depts/chemistry/courses/toolkits/380 This site contains many references to online resources.

http://www.stolaf.edu/people/hansonr/nmr/24-7 The OleNMR site. From this site you can run the NMR anytime day or night from anywhere on campus.

I'll also have available in the laboratory a set of reference books from my personal library that you can peruse at your leisure.

Course Structure

Basically, there will be three components of this course:

Seminars Each day at 8:15 AM in Sc 310. Tuesdays, Wednesdays, and Fridays I will present information and answer questions about the techniques used for structural analysis. Each Monday (except the first) a problem set will be due and discussed. Each Thursday the discussion will be based on what you have done in lab. You will take turns showing us what you have learned about your unknowns. This will be a good time to help each other and to see how the others are thinking about their problem.

Independent laboratory work Any time each day between the hours of 10:00 am and 5:30 pm. (You may pick your own hours, depending upon how things are going.) I will give you three unknowns. Your job will be to determine their identities over the course of the term. These will be relatively pure compounds. You will also analyze a mixture. In addition, at various times I will be demonstrating selected laboratory techniques, such as TLC and column chromatography, proper use of the rotovap, and use of the various instruments.

Problem sets and exam Three problem sets will be handed out. They will be due on Mondays, Jan. 12, 19, and 26. Each problem set will be worth 50 points. In addition, we will have an open-book take-home exam Friday, Jan. 30, that will be distributed at 8:00 AM and due back by 5:30 PM.

Grading

Chemistry 380 is a graded course. The breakdown will be as follows:

Unknowns:	150
I 40	
II 50	
III 60	
Medicinal Analysis	100
Problem Sets (3x50):	150
Final exam:	100
Total:	500

Chemistry 380 Seminar Schedule, January 2004

Date	Topic
Mon, Jan 5	Techniques for the identification of organic substances
Tue, Jan 6	Basics of infrared spectroscopy
Wed, Jan 7	Basics of NMR: chemical shifts
Thu, Jan 8	Discussion of Unknowns
Fri, Jan 9	Basics of NMR: coupling constants
Mon, Jan 12	Problem Set I due and discussed
Tue, Jan 13	Derivatization techniques
Wed, Jan 14	C-13 NMR and DEPT
Thu, Jan 15	Discussion of Unknowns
Fri, Jan 16	2-D NMR: COSY, HMQC, HMBC
Mon, Jan 19	Problem Set II due and discussed
Tue, Jan 20	Theory of Mass Spectrometry
Wed, Jan 21	Separation techniques
Thu, Jan 22	Discussion of Unknowns
Fri, Jan 23	TBD*
Mon, Jan 26	Problem Set III due and discussed
Tue, Jan 27	TBD*
Wed, Jan 28	TBD*
Thu, Jan 29	Discussion of Unknowns
Fri, Jan 30	Final Exam (distributed at 8:00 AM; due back at 5:30 PM)

* To be determined based on the interest and needs of the group.

Unknowns and Medicinal Analysis

You will be responsible for the analysis of three unknowns and one over-the-counter medicine. For the unknowns, your objective will be to propose the identity of the substances, and (except in the first case) to prove that identity by making a known chemical derivative of the substance. In the case of the medicine, your objective will be to separate its chemical components and analyze each of them fully.

The three unknowns will be as follows:

Unknown I: A pure substance with only one or two functional groups.

> To help you out on the first unknown, I will give you a sheet containing a list of several substances. Your unknown is one of the substances on this list. This should give you an idea of how ruling out a substance or functional group can be almost as valuable as identifying its presence. The basic objective here is to see how to use the reference books and online resources available to you and to reacquaint yourself with the equipment around the lab. Some may be new to you; others you have seen before, but have not actually sat down and tried to use yourself.

Unknown II: A second pure substance with only one or two functional groups.

> A similar problem, but now you won't have a list, making this one more difficult. After determining by solubility, IR, NMR, GC/MS, and perhaps a chemical test either what the compound is, or what sort of compound it must be, make a derivative, and confirm your proposal based on melting point and/or NMR spectroscopy of that derivative.

Unknown III: A pure substance with two or three functional groups.

> The only additional trick here is that your compound will have more than functional groups (COOR, OH, NH, C=C, etc.). Thus, when you look at the IR and NMR, you will have to sort out the information that is there more carefully than before (some may overlap).

For your fourth project, I'd like you to choose an over-the-counter medicine containing at least two active compounds. This could be a cold or allergy medicine, analgesic, or just about anything you think would be interesting. (You'll need to OK the choice of medicine with me prior to starting.) Your job will be to separate at least two of these compounds, purify them, and carry out an analysis of each. In addition, you should try to make at least one derivative. Here is where the NMR will come into full use. You should expect to carry out the full range of NMR experiments, including H-1, C-13, DEPT, COSY, HMQC, HMBC, and possibly some decoupling experiments. Since you will already know the chemical names of the compounds you are isolating, the primary objective will be to carry out the analyses with the small amount you have isolated and fully discuss them.

Reports

> Reports must completely describe the results of the analyses carried out. They need not go through the *logic* of the analysis (that is what the presentation is for). Instead, the report is to be a careful listing and interpretation of the data. Blank report forms can be found at the course home page.