

**CHEMISTRY 324W
ORGANIC LABORATORY**

Lecture: Monday and Friday 2:15-3:15; Reichardt building 204
 Lab: Wednesday (2:15-5:15) and Friday (3:30-6:30); Reichardt building 137
 Instructor: Tom Clausen, Reichardt building 188, 474-5512; FFTPC@UAF.EDU

Required Materials: Techniques in the Organic Laboratory, Pavia, Lampman, Kriz and Engel, Harcourt College Publishers.

8"x10" bound notebook . You can purchase one in the Chem office for about \$3.00 (our cost).

Recommended Materials: USB memory stick for backing up data and text files

Fees: \$120 Material fee for chemicals and other supplies
 \$45 Computer lab fee (charged only once for multiple chemistry classes)

Course Goals: This course emphasizes several aspects of organic laboratory practices. These include:

1. Synthetic procedures
2. Chromatographic analyses (primarily gc, column, HPLC, and tlc)
3. Standard work-up procedures
4. Purification techniques (crystallization, distillation, extraction, chromatography...)
5. Spectroscopic analyses (primarily NMR, MS and IR)
6. Literature searches
7. Scientific writing
8. Mechanistic studies
9. Use of relevant computer software
10. Chemical calculations including stoichiometry

Experiment sources: While the text for this course is an excellent guide to the techniques used by organic chemists, it is not a source of standard organic laboratory experiments. Rather, the experiments performed in this course will come from the previous week's handouts. In many cases these handouts will be taken from the chemical literature and will describe experimental details of syntheses **related** to the actual ones we will pursue. Consequently, the precise details of the procedure you will follow will be different; reagents, reaction times, reaction scale, apparatus setup, and scale of the experimental may be modified. This approach will provide you with the experience of using procedures from chemical literature as templates for designing your own synthetic strategies. *Because the details of each experiment will be developed in the lecture, it is imperative you maintain excellent attendance and be on time.*

The following references may be useful and should be found (and kept) in the Organic Teaching Laboratory:

- *Aldrich Chemical Catalog* gives physical properties as well as safety issues for most commercially available organic reagents.
- *The Merck Index* is an excellent reference book for over 10,000 important organic substances. It has a handy cross index and molecular formula index that you will find useful.
- *The CRC Handbook* is another reference book that provides some physical and spectral information on a wealth of substances. Overall, however, I find the Merck Index to be both easier to use and more relevant.
- *Organicum: Practical handbook of organic chemistry* by B.J. Hazzard (1973) provides useful experimental details for a number of standard organic reactions. This text is out of print.
- *Advanced Organic Chemistry: Reactions, Mechanisms, and Structure* by March (McGraw-Hill) is particularly useful because it provides good references to the chemical literature.
- *The Chemist Companion: A handbook of practical data, techniques, and references* by A.J. Gordon and Richard A. Ford (John Wiley & Sons) is an good source of information for all chemists (inorganic, organic, analytical...).
- *Reagents for Organic Synthesis* by Fieser and Fieser, volumes 1-13 (John Wiley & Sons) has detailed discussions about nearly every organic reagent with references to the chemical literature. At times details about how the reagent is typically used in a given reaction is provided.

- *Organic Synthesis*; collective volumes 1-5 (John Wiley & Sons) provides **very** detailed procedures for specific syntheses. The scale of the reactions, however, is usually large.
- *Spectroscopic Identification of Organic Compounds*; 6th Ed., by R.M. Silverstein, G.C. Bassler, and T.C. Morrill (John Wiley & Sons) provides good discussion and extensive tables for the interpretation of standard IR, H NMR, C NMR, and mass spectra. More advanced topics such as 2-D NMR and NMR of other nuclei are also discussed.
- *Compendium of Organic Synthetic Methods* (Vol I-III) provides specific examples (with reference) of functional group transformations. No discussion, however, is provided for these reactions.
- *Comprehensive Organic Transformations* is similar in content to *Compendium of Organic Synthetic Methods* but the organization and examples are different.

There are also a number of on-line sources of information (that are assessable from the computer lab):

- WWW.HAZARD.COM is a good on-line source of Material Safety Data Sheets (MSDS). The department also keeps a set of MSDS in NSF 139.
- SciFinder Scholar is one of the best ways to do a fast, easy, and thorough literature search.
- ACD labs is useful in predicting NMR spectra (based on model compounds) and naming organic compounds.
- HyperChem is a powerful tool for molecular modeling studies
- Mestrec is a useful program for viewing your NMR data on PCs and pasting spectra into reports.

Laboratory Safety: Laboratory safety is a major concern of all chemical laboratories but is especially important in organic labs due to the presence of flammable solvents, potentially hazardous fumes, highly reactive reagents, etc. The first lecture will deal explicitly with these hazards and the appropriate safety measures you must follow. Subsequent lectures, besides covering the theory and pitfalls of the coming weeks' experiments and perhaps helping you interpret the previous week's experiment, will also cover specific hazards that you may encounter. Please attend these lectures and be prepared for the lab by doing any assigned readings and having your notebook prepared before coming to lab. If you are not prepared for lab you may be asked to leave.

Course requirements and points to consider:

I. Some laboratory experiments will require a formal report. **It is greatly preferred if the report is sent to me electronically** The report should be prepared on a word processor equipped with a spell checker. Chemical structures should be drawn using computer software (Chem Draw, IsisDraw, or ACD ChemSketch – the latter two are free downloads from the internet) all of which are installed on Chem Computer Lab workstations. Chemical structures, reactions, and mechanisms should be inserted directly into the text. Supplementary material (such as spectra) should be embedded into the document so that they are easily viewed. The report format is the usual chemistry format: Introduction, Methods, Results, Discussion, References and Acknowledgements. The following information should be included *if appropriate*.

- Introduction: What were the goals of the experiment? Be sure to clearly state any hypothesis.
- Methods: Be succinct, but do not leave out important details. We will learn to write these by reading some from the original literature.
- Results:
 - The percent yield along with an estimation of product purity by spectroscopic and/or chromatographic analysis.
 - Spectra (usually IR and/or NMR) along with their interpretation which means writing out descriptions of where the peaks are, and which atoms or groups caused those peaks, and why you made these assignments. In particular, evidence for the presence or absence of any possible contaminants (F) should be addressed by a detailed examination of the spectra, using reference spectra when available. Be sure to include a discussion of your integration results for the NMR data. Don't forget to show how your spectra verify the absence of likely impurities due to the absence of critical peaks.
 - Gas chromatographic traces, if required, should be included and peak identification should be attempted.
- Discussion:

- a. The balanced chemical equation(s) along with a detailed mechanism using curved arrows should be included. Indicate reversible and irreversible steps. In a separate paragraph, EXPLAIN what is happening in each step.
 - b. Provide a summary of the possible contaminants in your isolated product. Usually you can omit most inorganic substance (NaCl, Na₂SO₄, HCl- but NOT water) and highly reactive reagents such as Grignards that wouldn't survive workup conditions. Examples of likely contaminants include solvents, starting material(s), water, and side products. Describe how the experimental procedure minimized the formation of these contaminants (for instance the use of a large excess of one reagent will tend to consume the limiting reagents).
- E. Acknowledgements of any student whose data is used in your report but please do not acknowledge the teaching assistant or professor.
 - F. References. Be sure to never include a reference without refereeing to it somewhere in the text.

II. **Writing Intensive designator.** A description of what a writing intensive course such as this is found on the university web-site: www.uaf.edu/uafgov/faculty/cd/coreguide.html. In brief, the W designator means that a majority of your grade is based on your written work, that some of the work will be resubmitted with revisions to previous comments and that factors such as content, organization, tone, word choice, grammar, spelling, sentence structure, etc., can contribute to the final grade. Please note that the prerequisite for all W courses is Engl 111X and Engl 211X or 213X.

While you are encouraged to collaborate with your classmates in interpreting your data and proofreading your reports, it is essential that you write your reports independently. Each paragraph should portray your own creativity and not simply paraphrase someone else's writing. It is a common misconception that changing the word choice, sentence structure or organization of an existing document protects against a charge of plagiarism; it does not.

You may use another student's figures in a report if such a move is (1) approved by the other person, and (2) proper credit is given to the individual who created them.

III. For some experiments, you will also hand in your chemical product. Put the compound in a vial with a piece of foil as a cap-liner to prevent contamination by the cardboard cap-liner. Store in the refrigerator or freezer until it is time to hand it in to prevent evaporation or degradation. Always label the vial neatly with your name, date, compound, mass and mp or bp. **Poorly labeled vials may be disposed of by chemistry personnel during routine cleanups. NEVER DISPOSE OF YOUR PRODUCT UNTIL YOUR REPORT HAS BEEN GRADED!!**

IV. Maintain an up to date notebook. Before each lab you should enter (i) a balanced chemical equation, (ii) a procedural outline or flow chart, and (iii) physical and hazardous properties for each chemical (including solvents) you plan to use in the experiment. Obtain this information from the Web – for example, if you look up the compound at the Aldrich chemical Co web site- <http://www.sigmaaldrich.com/catalog/search/AdvancedSearchPage> then follow the link to MSDS, the Material Safety Data Sheet will provide the appropriate information (Please keep in mind that the MSDS info is geared to handling chemicals on the industrial scale).. During the lab make notes on (i) your actual procedure including weighing information, (ii) significant visual observations, (iii) TLC sheets taped in, including solvent info, and (iv) spectra or references to location of spectra in a separate collection (I strongly recommend you enter file names of spectra you run directly into the notebook).

V. Reports and products are due on Fridays by 6:30 PM two weeks after the scheduled lab work. This is considered ample time for you to complete the project. Late reports may result in a reduction of 5% of the allowable points per day late!

Notebooks will be collected several times during the semester and returned graded by the next lab period. Details about what constitutes a good notebook will be provided in another document. One aspect of a good notebook will be mentioned here: A person should be able to write a report or repeat your experiment based solely on the notebook and any references provided in it.

VI. The final weeks of laboratory will be devoted to a "Research Project". Using methods from the text, you will try to solve a problem such as devising a synthesis or determining a mechanism. Your final

Week of	Topics	New Techniques	Readings
Jan 26	Exp 1: Spectroscopic Analyses of unknowns; Safety; Check-In; Review of computing facilities	IR Sample Prep NMR Safety Laboratory Notebook	Chapter 2 26.1 26.4-26.7
Feb 2	Exp 2: Stereospecificity in the hydride reduction of t-butylcyclohexanone or camphor	Computational Chemistry NMR Coupling Constants Anisotropy Effects effects in NMR TLC Extraction & Drying Agents	Chapter 29 26.10 26.8 Chapter 20 Chapter 12
Feb 9	Exp 3: Mass spectral analyses of spice using SPME and steam distillation of a spice.	Distillation Extraction Gas Chromatography	Chapter 18 Chapter 12 Chapter 22
Feb 16	Exp 4: Fisher Esterification using acetic acid and 2-methyl-1-butanol	Reflux Simple Distillation	Chapter 7 Chapter 14
Feb 23	Exp 5: Grignard addition to Benzoin-Diastereomeric excess	2-D NMR	
March 2	Exp 5 (cont); Acetonide Formation & Exp 6: Aldol Condensation	Recrystallization melting point determination	Chapter 11 Chapter 9
March 9	Spring Break- No classes		
March 16	Exp 7: Lichen extraction; isolation of usnic acid	HPLC	
March 23	Exp 8: Diels-Alder Reaction		
March 30	Start Research Project Synthesis of meso and racemic 3,4-di-C-carboxy-2,5-dideoxyhexaric acid		
April 6			
April 13			
April 20	End research project; No class on Friday		
April 27	Lab Clean-Up and Poster presentation		

Checklist for running spectra:

Experiment Name: _____

Date sample submitted: _____

Name of Data File _____

HNMR CNMR HSQC HMBC HSQC COSY DEPT

IR/Nujol IR/Neat IR/KBr

MS (Circle one)

Solvent (NMR or MS only)

What SPECIFIC question(s) are you attempting to answer by obtaining this spectrum?

Structure of major anticipated component of the sample:

Expected key features of the spectra for the anticipated product:

Expected *key* features of the spectra for each of the possible impurities. Describe only those aspects that should be easily seen in the spectra. Avoid commenting of ambiguous observations such as absorbances that would be masked by other expected compounds.

solvent(s)

Starting materials

Side Product(s) (likely ones only)

Attach any computer generated spectra (such as ACD Lab) that support your expectations