

CHEMISTRY 324W
ORGANIC LABORATORY

Lecture: Monday 2:15-3:15 Reichardt 204
Lab: Wednesday, 2:15-5:15; Friday 3:30-6:30 Reichardt 137
Instructor: Thomas Green, Reichardt 174, 474-1559; ffitkg@uaf.edu
Office Hours: Tues, Thurs 2-5

Required Materials: Techniques in the Organic Laboratory, Pavia, Lampman, Kriz, Engel, 2002, Harcourt
8"x10" bound notebook, safety glasses

\$10 Key deposit for lab drawer keys and locker keys, to be returned at check-out, minus any appropriate glass breakage costs

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

1. Synthetic procedures
2. Chromatographic analyses (primarily gc, cc, tlc)
3. Standard work-up procedures
4. Purification techniques (crystallization, distillation, extraction, chromatography...)
5. Spectroscopic analyses
6. Literature searches
7. Scientific writing – **all experiments require a written report.**
8. Mechanistic studies
9. Poster Presentations

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. It is hoped that 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 sustain excellent attendance.*

The following references may be useful:

- *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, ¹H NMR, ¹³C NMR, and mass spectra. More advanced topics such as 2-D NMR and NMR of other nuclei are also discussed.

- 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.

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, Writing Requirements, and points to consider:

I. **A written report is required for all experiments.** The report should be typed on a word processor equipped with a spell checker. Chemical structures should be drawn using computer software (Chem Draw or Isis Draw). The following information should be included if appropriate.

- A. The **chemical reaction** along with a detailed **mechanism** using curved arrows. Indicate reversible and irreversible steps.
- B. A list of likely **contaminants** in the isolated product. These contaminants should include probable side products as well as solvents and unreacted starting materials.
- C. A detailed flow diagram showing your **separation procedure**. See p 25 text for example.
- D. The **percent yield** along with an estimation of **product purity** by spectroscopic and/or chromatographic analyses.
- E. **Spectra** (usually IR or NMR) along with their interpretation. 1-D NMR (¹³C and ¹H) spectra should be submitted. Structures should be drawn on the spectrum with assignments made. Impurities and solvents should be identified where possible. Varian file names for 2D spectra should be indicated in the report as varian *.fid files so that I can access these files when needed. IR and MS spectra should be scanned and submitted as an image file (gif, jpeg).
- F. **Gas chromatographic traces**, if required, should be included and peak identification should be attempted.
- G. **Acknowledgments** towards any student whose data is used in your report.

II. Submit your product to the TA in a small vial, labeled with your name, product name, and mass.

III. 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 MSDS sheets located in room 139). During the lab make notes on (i) your actual procedure including weighing information, (ii) significant visual observations, (iii) sketched TLC plates, including solvent info, and (iv) references to location of spectra (file names!) in a separate collection.

IV. **Reports and products are due according to the attached schedule.** Late reports may result in a reduction of 5% of the allowable points per day late! Attach spectra as discussed above in IE. All experiments require a written report but two reports (Grignard and Diels-Alder) will be evaluated and **returned for your revision**. All reports are worth 50 pts except for the Grignard and Diels-Alder reports (75 pts each).

V. Notebooks will be collected at the midterm and at the end of the semester for grading

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**

report will be in the form of a poster presentation and an oral presentation. Each student must meet with the instructor for a review of the poster, prior to presentation.

Lectures. It is essential that you attend all lectures and arrive on time to the laboratory in order to fully understand the experiment and safety issues.

Each lecture will begin by pointing out salient features for the upcoming experiment. Questions regarding 1) the choice of solvent, 2) order of addition, 3) which reagent to use in excess, 4) work-up steps, 5) appropriate stopping points, etc., will be addressed. In addition, as time allows, other topics will be covered that are described in the syllabus. Much of your midterm and final will come from these lectures. In addition, special topics (to be announced) will be presented during the research project phase of the course.

Most experiments will be significantly modified from the description given in the handouts. These modifications may include 1) reduction in the scale of the reaction, 2) changes in the glassware used, 3) additional analyses of the final product, and 4) alternative reagents, solvents or starting materials. A discussion of these modifications will be presented in the lecture.

Finally most experiments pose specific hazardous operations that will be detailed during the lecture and at the beginning of the laboratory.

Grades: The final letter grade will be based on the total number of points accrued during the semester, apportioned as follows:

Experiments 7	400
Research Poster	100
Oral Presentation	50
Notebook	50
MidTerm	100
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	700

Plus/minus Grades will not be assigned in this class.

Grading Scale:

90-100% A

80-89% B

70-79% C

60-69% D

<60% F

Disability Statement (as required by UAF policy)

"Students with a physical or learning disability are required to identify themselves to Mary Matthews in the Disability Services Office, located in the Center for Health and Counseling in order to receive special accommodations. The students must provide documentation of the disability. Disability Services will then notify the course instructor of special arrangements for taking tests, working homework assignments, and work in the laboratory."

Honor Code Statement and Departmental Policy on Cheating (as required by UAF policy)

Students are referred to the official university policy, as given on pages 24-25 in the University Catalog. Consider the following excerpts:

"Students will not collaborate on any quizzes, in-class exams, or take-home exams that will contribute to their grade in a course, unless permission is granted by the instructor of the course. Only those materials permitted by the instructor may be used to assist in quizzes and examinations.

Students will not represent the work of others as their own. A student will attribute the source of information not original with himself or herself (direct quotes or paraphrases) in compositions, theses, and other reports.

No work submitted for one course may be submitted for credit in another course without the explicit approval of both instructors.

Violations of the Honor Code will result in a failing grade for the assignment and, ordinarily, for the course in which the violation occurred. Moreover, violation of the Honor Code may result in suspension or expulsion."

Instructor Withdrawals: The instructor reserves the right to withdraw any student from class if that student has missed an exam without an excused absence, has missed more than two labs, or appears to be failing as of March 12 - the last day for instructor-initiated withdrawals.

Incompletes: A grade of “incomplete” is assigned only when a student misses the final exam for an acceptable reason, such as a medical problem, a death in the family, etc. Students must address an incomplete grade in the semester immediately following that for which it was assigned.

LABORATORY SAFETY RULES

1. Do not begin any procedure until you understand the theory and technique to be used. This includes familiarizing yourself with hazardous properties of all chemicals and solvents before use.
2. As you plan your work try to anticipate potential safety hazards and prepare for them. For instance, if an operation may result in a fire, make sure flammables are not stored nearby.
3. If in doubt about safety hazards, consult the instructor or teaching assistant before proceeding.
4. Safety glasses or goggles must be worn at all times. Contact lenses are generally not allowed.
5. Shoes and appropriate dress are to be worn at all times. Secure long hair and loose sleeves. Shorts and sandals are not appropriate.
6. Familiarize yourself with the location and operation of fire extinguishers, safety showers, eyewash fountains, spill sorbents, and other safety devices in and near the lab.
7. Coats, books and other personal items must not restrict easy access around the lab. The best place for such items is on the rack outside the lab.
8. Food, beverages, smoking and open flames are not permitted in or near the lab.
9. Keep floors and work surfaces clean and dry. Clean up any spills immediately.
10. Unauthorized procedures are not permitted.
11. Report all accidents or unsafe conditions to the instructor.
12. Laboratory work at other than scheduled class times is not allowed.
13. Attend all lectures and arrive to lab on time so that you will be aware of specific hazards.
14. Do not dispose of chemicals down the sink or in the normal trash. Waste containers will be provided. Keep the cap on the waste containers in place and record on the waste bottle label the identity and amount of each discarded waste.
15. Do not let friends or family visit you in the lab.
16. Wash your hands regularly within the lab. This is especially true upon leaving the lab.
17. If you come in physical contact with a chemical, wash the effected area will large amounts of cold water and soap immediately. Remove highly contaminated clothing promptly.
18. No open flames (including Bunsen burners) are allowed in the organic lab.

Schedule for Fall 2008
Chem 324 Organic Laboratory

Week of	Topics	Techniques	Readings
5- Sept	Fri introduction		
8-Sept	Check in, safety lecture; computer lab. Exp 1: Spectroscopic Analysis of Unknown	Safety, Laboratory Notebook, IR, NMR Sample Prep	Ch 1,2 25.1,25.2 26.1, 26.4-26.7
15-Sept	Exp 2: Camphor Reduction	Computational Chemistry, Extraction & Drying Agents	Ch 12, 29, 26.9-26.11
22-Sept	Exp 3: Esterification of Benzoic acid; Nitration	Distillation	Ch 14.1 - 14.3
29-Sept	Catch up - write reports Submit Research Plan	No Lab	
6-Oct	Exp 4: Fragrance analysis- MS fragmentation	Gas chromatography-mass spectrometry; computational NOE NMR, stereochemical analysis.	Ch 28 Ch 7
13-Oct	Exp 5: Grignard Reaction		
20-Oct	Exp 5 (cont)		
27-Oct	Mon: review	Friday: Midterm (no lab)	
3-Nov	Exp 6: Lichen Extraction	Recrystallization, melting point	Ch 9,11
10-Nov	Exp 7: Diels-Alder Rxn	2-D NMR analysis	Handout on 2D NMR
17-Nov	Exp 8: Research project		
24-Nov	Exp 8: Research project		No class Friday
1-Dec	Exp 8: Research project	Thursday - Dec 4 Dept Poster	No class Friday
8-Dec	Catch up -write reports	No Lab	
17-Dec	Final: 12 min oral presentations of Research Projects		

Due Dates for Lab Reports

Expt 1 (Spectroscopic Unknown) Sept 29
 Expt 2 (Camphor) Oct 6
 Expt 3 (Esterification/Nitration) Oct 13
 Expt 4 (Mass Spectrometry) Oct 27
 Expt 5 (Grignard Reaction) Nov 10
 Expt 6 (Lichen extraction) – Nov 24
 Expt 7 (Diels-Alder) Dec 1
 Expt 8 (Research) Poster Dec 11

Expts 1 – 4 and Exp 6 are short reports – 50 pts each x 5 = 250 pts
 Expt 5 and 7 are full length narrative reports. – 75 pts each x 2 = 150 pts
 Expt 5 and 7 reports will be submitted for instructor review and returned for editing
 before submitting a final version.