Lecture: ...................Monday and Friday 2:15-3:15; Reichardt 204
Lab: .........................Tues and Thurs., 2:00-5:15; Reichardt 137
.................................Wed 2:00-5:15 and Fri 3:30-6:30; Reichardt 137
Instructor: ...............John Keller, Office 161 Nat Sci, 474-6042; jwkeller@alaska.edu
.................................Office hours by appointment, or drop-in.
Teaching Assistants  Zhipeng Dai  zdai@alaska.edu
               Jamie McKee  jamckee@alaska.edu

Required Materials: (1) PAVIA et al. , MICROSCALE+MACROSCALE TECH.IN
ORGAN.LAB , $112 new at the UAF bookstore, $84 used. Amazon.com:
$106 new, with free shipping, $48 used + $4 shipping
(2) Hayden-McNeil spiral bound notebook ($10 from Keller)

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

Fees: (1) Material fee for chemicals, glassware breakage, and other supplies $120
(2) chemistry computer lab fee $45 (charged only once for multiple chem classes)
(3) key deposit $5 cash (Bring it to first lab.)

Course Goals: This course emphasizes several aspects of chemistry laboratory practices. These
include:
  1) Synthetic procedures
  2) Spectroscopic analyses (NMR, MS, and IR)
  3) Standard work-up procedures
  4) Scientific writing
  5) Use of relevant computer software
  6) Chromatographic analysis (gc, tlc)
  7) Purification techniques (crystallization, distillation, extraction, chromatography...)
  8) Literature searches
  9) 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 will come from handouts or directly from the chemical literature. In some cases the
handouts will describe experimental details of syntheses related to the actual ones we will pursue.
Consequently, the precise details of the procedure you will follow may differ in terms of the
reagents, reaction times, reaction scale, apparatus setup, and scale of the experimental. This
approach will provide you with the experience of using procedures from the chemical literature as
templates for designing your own synthetic strategies. Because the details of each experiment will
be explained in lecture, it is imperative you attend (and be on time).
**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 lab (Tuesday or Wed) will deal explicitly with these hazards and the appropriate safety measures to follow. Subsequent lectures, besides covering the theory and practicalities of the next 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:**

I. A written report is required for each experiment. Some will be shorter, others longer.

Other details:

1. Prepare using Microsoft Word, or compatible, software.
2. Please use the spelling and grammar checkers before handing anything in!
3. Submit in both hard copy (stapled) and electronic form, formatted identically. The hard copy should be printed on the Kyocera Color Laser (room 172) or similar printer and placed in the TA’s mailbox in the Chemistry office 194 NSF, or in the provided strongbox. The electronic copy should be emailed to your TA.
4. Chemical structures can be draw neatly by hand or using computer software (ChemWindow, ACD ChemSketch - the latter is free download from the Internet, or MarvinSketch). Chemical structures, reactions, and mechanisms should be inserted directly in the text, not at the end.
5. IR and NMR spectra should be pasted as graphics in a separate section of "Figures" at the end of the report.
6. The report format is the usual one used in the sciences: Introduction, Methods, Results, Discussion, Acknowledgments, References. Include figures such as NMR spectra at the back.
7. Formatting: Use 12-pt Times Roman font, single space, margins 1.5" all around.

Some notes on the different sections of chemistry reports:

- **Introduction:** Describe the chemistry goals for the experiment. Write a balanced chemical equation for the reaction, if any.
- **Methods:** Describe your procedure in passive voice, third person language. Be succinct, but do not leave out important details. We will learn how 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 analyses.
- **Results:** 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 assigned those atoms. In particular, evidence for the presence or absence of any possible contaminants should be addressed by a detailed examination of the spectra, using reference spectra when available. Be sure to include a discussion of the integration analysis of the $^1$H-NMR spectra.
✓ NB. Please **do not refer to or display NMR spectra calculated by ACD software anywhere in the report.** Use the data in the spectrum itself, and if necessary, chemical shift or frequency correlation tables from your text.

✓ Results: Gas chromatographic traces, if required, should be included and peak identification should be attempted.

✓ Discussion: A detailed mechanism using curved arrows. Indicate reversible and irreversible steps. Good idea to label intermediate structures (1, 2, 3) then in a separate paragraph, explain what is happening in each step.

✓ Discussion: if there are contaminants in the isolated product, point out how the experimental procedure minimized the formation of any of these contaminants (for instance the use of a large excess of one reagent will tend to consume the limiting reagents).

✓ Acknowledgments of any student whose data is used in your report. **Do not** acknowledge the teaching assistant or professor.

✓ References used. Include a reference to the original literature in each formal report. If possible, use the Endnote application available in the Chemistry Computer Lab. Never include a reference without a reference to it (a “callout”) appearing somewhere in the text.

II. **Writing Intensive designation.** 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 based on previous comments, and that factors such as content, organization, tone, word choice, grammar, spelling, sentence structure, etc., contribute to the final grade. The prerequisite for all W courses is Engl 211X or 213X.

While you may collaborate with classmates in interpreting data and proofreading 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.

**Reaction Schemes in Reports.** You may use another student’s formulas in a report if such a move is (1) approved by the other person, and (2) proper credit is given in the Acknowledgements to the individual who created them.

III. For most 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). Always label the vial neatly with your name, 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.** Store in the refrigerator or freezer in 139 Reic until it is time to hand it in to prevent evaporation or degradation.

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](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.*) We will in some labs also have pre-lab questions that will be due at the beginning of lab. During the lab make notes on (i)
your actual procedure including weighing data, (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. DATE each page or entry.

**Notebook sheets** will be collected at the end of lab each Thursday or Friday.

V. Except for Expt 1, reports and products are due on Fridays by 6:30 PM generally about two weeks after the scheduled lab (see the lab schedule). *Late reports will not be accepted – hand in what you’ve got it’s better than a zero.*

VI. The final weeks of laboratory will be devoted to a “Research Project”. This will involve trying to solve a problem such as devising a synthesis or determining a mechanism. Your final report will be an article in the style of the *Journal of Organic Chemistry*.

**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 the midterm exam will come from these lectures.

Some experiments may have to be 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 and possibly at the beginning of the lab itself. *This is why lecture attendance is mandatory.* Also, always check the **marker board in lab** (137 NSF) for important announcements.

**Grades:** The final letter grade will be based on the total number of points accrued during the semester, apportioned as follows. (+/- grades may be used).

<table>
<thead>
<tr>
<th>Experiment</th>
<th>Points</th>
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<tbody>
<tr>
<td>Experiments 1, 2, 4, 6, 7, 9 (50 pts each)</td>
<td>300</td>
</tr>
<tr>
<td>Experiments 3, 5, 8 (100 pts each)</td>
<td>300</td>
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<tr>
<td>Project</td>
<td>200</td>
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<tr>
<td>Midterm</td>
<td>100</td>
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<tr>
<td>Final</td>
<td>100</td>
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<tr>
<td>Notebook</td>
<td>100</td>
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<tr>
<td><strong>Total</strong></td>
<td><strong>1100</strong></td>
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**Students with documented disabilities** who may need reasonable academic accommodations should discuss these with me during the first two weeks of class. You will need to provide documentation of your disability to Disability Services in the Center for Health and Counseling, 474-7043, TTY 474-7045.
<table>
<thead>
<tr>
<th>Week</th>
<th>Week of</th>
<th>Topics</th>
<th>New Techniques</th>
<th>Readings</th>
<th>Report due</th>
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</thead>
<tbody>
<tr>
<td>1</td>
<td>18-Jan</td>
<td>Fri introduction</td>
<td></td>
<td>Chapter 2, 26.1, 26.4-26.7 Text NMR,IR</td>
<td></td>
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<tr>
<td>1</td>
<td>24-Jan</td>
<td>NMR theory &amp; practice: Check in, safety lecture;, S5</td>
<td>Safety, Laboratory Notebook, NMR Sample Prep</td>
<td>Chapter 2, 26.1, 26.4-26.7 Text NMR,IR</td>
<td>31 Jan/1 Feb</td>
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<tr>
<td>2</td>
<td>31-Jan</td>
<td>Exp 1: Spectroscopic Analysis of Unknown 1</td>
<td></td>
<td>Chapter 12, 20, 29, 26.10, 26.8</td>
<td>18-Feb</td>
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<tr>
<td>3</td>
<td>7-Feb</td>
<td>Exp 2: Spectroscopic Analysis of Unknown 2</td>
<td>IR spectroscopy</td>
<td>Ch 18, 12, 22</td>
<td>25-Feb</td>
</tr>
<tr>
<td>3</td>
<td>7-Feb</td>
<td>Exp 3: Stereospecificity in Metal Hydride-Ketone Reduction</td>
<td>Computational Chemistry, NMR Coupling Constants, Anisotropy effects in NMR, Extraction &amp; Drying Agents</td>
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<tr>
<td>4</td>
<td>14-Feb</td>
<td>Exp 4 Fragrance analysis-MS fragmentation</td>
<td>Gas chromatography-mass spectroscopy</td>
<td>Chapter 7,14</td>
<td>4-Mar</td>
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<tr>
<td>5</td>
<td>21-Feb</td>
<td>Writing labs 3 &amp; 4</td>
<td></td>
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<tr>
<td>6</td>
<td>28-Feb</td>
<td>Exp 5: Fisher Esterification</td>
<td>Simple Distillation</td>
<td>Chapter 9,11</td>
<td>25-Mar</td>
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<td>7</td>
<td>7-Mar</td>
<td>Exp 6 Aldol</td>
<td>Recrystallization, melting point Midterm Exam (Friday Mar 11)</td>
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<td>1-Apr</td>
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<td></td>
<td>14-Mar</td>
<td></td>
<td>Spring break</td>
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<td>8</td>
<td>21-Mar</td>
<td>Exp 7: Diels-Alder Library</td>
<td>SciFinder Scholar</td>
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<td>8-Apr</td>
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<tr>
<td>9</td>
<td>28-Mar</td>
<td>Exp 8 Cu/SnCl2 allylation</td>
<td>gNOESY</td>
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<td>15-Apr</td>
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<td>10</td>
<td>4-Apr</td>
<td>Exp 9: TBA</td>
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<td>22-Apr</td>
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<tr>
<td>11</td>
<td>11-Apr</td>
<td>Exp 10: Project</td>
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<td>13-May</td>
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<tr>
<td>12</td>
<td>18-Apr</td>
<td>Project</td>
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<td>13</td>
<td>25-Apr</td>
<td>Project</td>
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<tr>
<td>14</td>
<td>2-May</td>
<td>Project</td>
<td>Check out thurs/fri</td>
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<tr>
<td>15</td>
<td>9-May</td>
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<td>Final Exam 1-3 PM Monday, May 9</td>
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References

The following references may be useful and should be found in (and should not be removed from) the laboratory:

- **Aldrich Chemical Catalog** gives physical properties as well as safety issues for most commercially available organic reagents. (You can order your own free.)
- **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. (The Merck Index is easier to use and more relevant.)
- **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.

- **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 – literature searching
- ACD labs NMR software
- HyperChem software for molecular calculations
- Japan spectral database   [http://riodb01.ibase.aist.go.jp/sdbs/](http://riodb01.ibase.aist.go.jp/sdbs/)