Text: The text is The Student’s Lab Companion. Laboratory Techniques for Organic Chemistry, by John W. Lehman, Pearson Prentice Hall, 2nd edition 2008, ISBN 0-13-159381-1 OR 1st edition, 2004, ISBN 0-13-017867-5. The text is optional. The experimental procedures are on the course website, accessible through Moodle at https://moodle.oakland.edu. Alternatively you can purchase a coursepack with all the experiments and other documents at Textbook Outlet, Bin #8002. You also will need a bound laboratory notebook (preferably grid-lined) and safety goggles suitable for work with chemicals. You can choose to use a lab book with duplicate carbon pages instead. I will only accept lab reports that have been written in a lab book – no computer written reports.

This course covers basic organic laboratory manipulations at the semi-micro and micro level, syntheses, spectroscopy, and chromatography, intended for all students who need experience in organic chemical techniques. Goals for the class include:

- Learn how to perform a variety of methods for identification of organic compounds and determination of purity. These include melting point, boiling point, refractive index, IR and $^1$H-NMR spectroscopy, and thin-layer chromatography, as well as chemical tests for the presence of functional groups.
- Learn a variety of laboratory techniques for isolating and purifying compounds, including extraction, washing, distillation, recrystallization, and trituration.
- Gain experience with microscale equipment and other laboratory equipment.
- Gain experience in several different reactions to synthesize compounds.
- Be able to properly record data, keep a laboratory notebook, and carry out calculations such as percent yields and a partition coefficient.

Your grade will be determined by 8 lab reports and a final exam. The lab reports for one day labs are worth 30 points while the ones for two days are worth 50 points each. There is also a final exam worth 60 points. At the end of the semester, all points will be added together. The total number of points is 360. You will be penalized for late reports if there seems to be a pattern of turning them in late. Anybody who gets at least 70% of the points will be guaranteed at least a 2.0. The grading scale is:

<table>
<thead>
<tr>
<th>Percentage</th>
<th>Grade</th>
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<tbody>
<tr>
<td>91-100%</td>
<td>4.0</td>
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<tr>
<td>89-91%</td>
<td>3.9</td>
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<td>88-89%</td>
<td>3.7</td>
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<td>86-88%</td>
<td>3.5</td>
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<tr>
<td>84-85%</td>
<td>3.4</td>
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Then 0.1 point less for each percent less, e.g., 80% is 3.0, 81% is 3.1, 77% is 2.7, etc.

The final exam is Tuesday, August 18, from 12 to 3 PM in room 200 of Dodge Hall. If you miss it, you must notify me by August 19 to be allowed to take a make-up.
Lab reports are due at the end of your lab period on the following dates:

- Experiments 1 and 2: July 15/16
- Experiments 3 and 4: July 22/23
- Experiments 5 and 6: August 5/6
- Experiments 7 and 8: August 16 at 5 PM

Once you turn in a lab report for grading you cannot make changes or complete missing parts or turn in missing samples or spectra. However, you may choose to repeat experiments if time permits before you turn them in for grading. Labs that you are unable to complete before the due date can be turned in on the next due date. At the end of the semester, you will be penalized for not disposing of samples that you turned in that were graded.

All labs must be in by Thursday, August 13, 2009 at 5 PM. They may be turned in earlier. If you turn them in between Thursday, August 13 at 5 PM and Tuesday, August 18, 2009 at 12 PM, you may lose points. You will lose 3 points on each lab turned in at the beginning of the final exam unless you have already arranged to turn the labs in at that time. After you start the final exam, any missing lab will receive a grade of zero. You **MUST notify me prior** to the deadlines if you must be late with the lab reports for any reason, no matter how excellent the reason is – the reason for being late is less important than the necessity of notifying me that your report will be late.

Make up labs can be done during labs other than on your usual day, starting the day the experiment is first run. There is also a makeup time the last week of class. Note that some experiments require drying of samples and cannot be done in one day.

Please turn off cell phones or pagers in class, or turn to vibrate mode. You may have points deducted if your cell phone or pager can be heard going off in class.

Students are expected to display academic integrity, as mentioned in the Student handbook.

**SAFETY INFORMATION**

The safety manual for the course is online on the Moodle site for the course. You need to read it over and click at the end that you are taking responsibility for knowing the material in it. You also will need to pass a safety test that is on Moodle. The test can be taken as many times as needed in order for you to pass it. **Acknowledging the Safety Manual and passing the test must be completed before you start working in lab on the second day of lab (July 6th or 7th).**
LECTURE SCHEDULE - CHEMISTRY 237 SUMMER 2009

Date         Lecture

June 29/30   Expt. 1. Extraction/Recrystallization
July 6/7     Expt. 2. B.P. and Fractional Distillation/Exp. 3 Eugenol
July 13/14   Expt. 3. Eugenol, start Expt. 4. Grignard
July 20/21   Finish Expt. 4, Grignard, and Expt. 5, p-Anisalacetophenone
July 28/29   Expt. 6, Nitration of methyl benzoate, and
             Expt. 7 Methyl Salicylate
August 3/4   Finish Expt. 7, start Expt. 8 ID Unknown Compound
August 10/11 Finish Expt. 8. ID Unknown Compound, and NMR

Tuesday, August 19, 12 to 3 PM
Final Exam 200 Dodge Hall

LABORATORY SCHEDULE

Date         Experiment                Lab Reference

June 29/30   Check In                  Handout
July 1/2     Extraction/Recrystallization Expt. 1
July 6/7     Extraction/Recrystallization Expt. 1
July 8/9     B.P. and Fractional Distillation Expt. 2
July 13/14   Isolation of Eugenol      Expt. 3
July 15/16   Grignard Reaction        Expt. 4
July 20/21   Grignard Reaction        Expt. 4
July 22/23   p-Anisalacetophenone     Expt. 5
July 27/28   Nitration of Methyl Benzoate Expt. 6
July 29/30   Methyl Salicylate        Expt. 7
August 3/4   Identification of an Unknown Compound Expt. 8
August 5/6*  Identification of an Unknown Compound Expt. 8
August 10/11/12/13 Make Up and Check Out

*Course Evaluations may be done on these days.

LABORATORY REPORTS AND NOTEBOOKS

You will need a bound notebook, preferably with graph-style paper, in order to write your lab reports and record data. The first page must contain a Table of Contents. Your name should appear on the outside of the book. At your option you can use two separate lab notebooks if you want to be able to work on some reports while the others are being graded. (If you choose this option, experiments 1,2,5, and 6 would be in one book and 3, 4, 7, and 8 in the other.) You may instead choose to use notebooks which give carbon-copies, where you either turn in the original or the copy, whichever is more legible. In either case, you must write your data and reports in the original lab book. I will NOT accept typed lab reports. In this class I am attempting to familiarize you with the professional way to write lab reports and keep a lab notebook, even though some to the legal reasons are not yet relevant. To properly document data, the date should be entered directly into a bound notebook with numbered pages, in ink. Spectra and machine printouts are taped or stapled in. Mistakes are crossed out, not erased or “whited-out.” With these practices, it would be obvious if data were altered or removed. Thus, the notebook contains the “true” data. The data page should be signed by either me or one of the teaching assistants. If you need to change the data afterwards get the changes signed by us also.
Refer to the sample Lab Book Write Up for the following comments. (I have not used graph paper in the interest of clarity of duplication.) Please put the appropriate items in the order listed.

The title is obvious, I hope. Note the page numbers. They do not start with “1” because there should be a Table of Contents at the very beginning if you are using a bound book.

The purpose states something about the techniques or type of reaction that you are learning. It is not necessary to mention the purification scheme, calculation of yield, etc.

The Table of Materials gives an indication of what reagents and other chemicals are used, how much will be needed, and why they are used. A table is the most readable way to write this section. Be sure to include structures. The amounts are for planning purposes; the exact amounts that you used belong in the data section. Glassware and instruments do not have to be listed. If you insist upon listing them, please put them at the end so that I can find the reagents.

The Reaction scheme shows the structures of reactants, products, and intermediates for which there is a separate step in the procedure, even if the intermediate is not isolated. However, you do not need to show transition states that are not stable, or detail the entire mechanism. Some experiments such as the distillation one do not involve a reaction.

The Procedure can be in paragraph or outline form and describes what you plan to do. It needs to be detailed enough for you to follow it. You must write the procedure in your lab book. Taping in a printed version is not acceptable. **You are not allowed to use the printouts of the procedures during the lab.** Consequently, if you have not prepared before you come you will not be able to do the experiment until you write out a procedure, and you will not be given more time at the end of class. I will only let you use printouts of the procedure for the first day of experiment 1. Afterwards you will be penalized if I catch you looking at printouts.

The Data Table can be prepared in advance so that all you have to do is fill in the blanks. However, you are not required to do so. I expect that all data and observations be entered directly into the book, not written on pieces of paper or the manual and transferred later. **You will lose points if I discover you writing data somewhere other than in your lab book.** By preparing the experiment in advance you will have all the space you need for the first four sections. I do not penalize for crossing things out and writing above. Unless a report is so messy that I can’t read it, neatness is not critical. Blank pages and writing on both sides of a page are both acceptable. Before you leave lab, have the instructor or one of the TA’s sign your data. We will not sign data pages written on loose sheets of paper. If you are waiting in line to use equipment, you can take cuts in front of someone who is planning to record data somewhere other than in his or her lab book.

Be sure to write your actual data in the Data section. For instance, if the lab procedure calls for 1.0 grams of a reagent, record the actual value that you measured in the data section. Writing “1.0 g” in the Table of Materials or Procedure is not adequate. Use all the decimal places that you can get from the equipment and include units, e.g., “Mass of benzoic acid, 1.000 g”, not “Benzoic acid 1.0”.
The Observations section is optional, if there is something you want to note for your own purposes.

The Calculations section includes any numerical calculations, including units. *These should not be put in the Conclusion section, or combined with Conclusions.* I don’t care if you do the calculations on separate paper and then copy them into your lab books, though you can do the calculations directly in the book. If you need to recalculate something it is perfectly OK with me if you cross out the old calculations. If you run out of room on one page, you can always continue on a later page with a page number for the continuation.

The Conclusion section concisely summarizes the requested results in one place. I do not like to search through the report to find the various pieces of information. When you have infrared spectra, NMR data, or GC data, attach them by staples or tape somewhere in the appropriate report in such a way that they do not cover the text and can be opened up easily. (The NMR spectrum in the sample report is of course not stapled as it would be hard to duplicate.) Be sure to mention if you have made significant changes in the procedure.

For experiments where the product (or a portion) needs to be turned in, I will show you where the vials are and where to turn them in. Once I have returned a report you can remove the sample from the holder and dispose of it properly by the end of the semester. For most samples this involves putting the solid in the crock and disposing of the vial in the wastebasket (for plastic) or broken glass containers. *You will be penalized for graded samples not removed by the end of the semester.*

Lab scores will be a combination of results and correct writing and calculations. Even absolutely atrocious results will not, in themselves, prevent you from passing the course, though not with a high grade. Experimental results will count as 20 to 25% of the grade. Results are graded for yield, purity (as determined by melting points, appearance, and IR spectra), or closeness to expected results (for unknowns and Extraction). Often, full credit for yield is give to the top 20 to 30% of the class and merely average yields are worth slightly less, so results are curved. *Once your report has been submitted for grading you cannot go back and recalculate data, submit missing samples or spectra, etc.* You may not redo an experiment that has been graded, but you may redo experiments that have not yet been submitted if you have the time.