CHE 171
Fall, 2005
Lab Guidelines & Schedule

In addition to good laboratory techniques and the methods of carrying out basic laboratory procedures, other things you will also learn from the laboratory portion of this course are:

- how to take data carefully
- how to record relevant observations
- how to use your time effectively
- how to assess the efficiency of your experimental method
- how to plan for the isolation and purification of the substance you prepare
- how to work safely
- how to solve problems and think like a chemist

(A) General Guidelines for the Organic Chemistry Laboratory:

1. Study the experiment and the reasons for each operation before you come to lab. Study, do not just read about the experiment before lab period. Although the techniques employed in the laboratory are not particularly difficult to acquire, they do demand a significant amount of attention. For you to reach a successful and happy conclusion, you cannot afford to have the focus of your concentration broken by having to constantly refer to the text during the experiment (this is why the texts are not allowed in the lab). Disaster is ever present for the unprepared.
2. ALWAYS work with clean equipment. You must take time to scrupulously clean your equipment before you start any experiment. Contaminated glassware will ultimately cost you additional time, and you will face the frustrations of experiencing inconsistent results and lower yields.
3. CAFEFULLY measure the quantities of materials to be used in the experiments. A little extra time at the beginning of the laboratory can speed you on your way at the end of the session. Many organic reactions are very sensitive to relative quantities of reagents. Do not be hurried or careless at the balance.
4. Clean means DRY. Water or cleaning solution can be as detrimental to the success of a reaction as dirt or sludge in the system.
5. ALWAYS work on a clean laboratory bench surface.
6. ALWAYS place reaction vials or flasks in a clean beaker when standing them on the laboratory bench.
7. ALWAYS think through the next step you are going to perform before starting it. Once you have added the wrong reagent, it is back to square one.
8. ALWAYS save everything you have generated in an experiment until it is successfully completed. You can retrieve a mislabeled chromatographic fraction from your locker but not from the waste container.
9. ALWAYS keep a permanent record of all lab work in a bound notebook.

(B) General Rules:

1. Safety goggles are to be worn at all times when in the laboratory; wear gloves when necessary.
2. No shorts, sandals or roller blades are to be worn in the laboratory.
3. Please read and be familiar with the University's Chemical Hygiene Plan.
4. Please respect the laboratory space and your classmates by cleaning up after yourself.
5. Please come to lab prepared by reading over the experiment prior to your laboratory period.

(C) Laboratory Notebooks:

Prepare your notebook AHEAD OF TIME according to the guidelines below. You will NOT be allowed to bring your textbook into the lab, so you'll rely solely on your notebook to get you through the experiment. Key components of a laboratory notebook include:

1. Date experiment was conducted
2. Title of experiment and reference for it
3. Purpose for running the reaction
4. Reaction scheme, a balanced equation (if applicable)
5. Table of reagents and product with data on the compounds' names, physical constants like MF, mp, bp (literature and experimental values), density, volume, weight and moles, theoretical and percent yield (where applicable), safety information related to exposure and toxicity from the Material Safety Data Sheets (MSDS)
6. Details of procedure and set-up used and all changes from procedure from the lab manual
7. Any observations you make while you're conducting the experiment
8. Characteristics of the products
9. Analytical and spectral data

Always record your data in ink. If a mistake is made, make a neat line through the word or words so they remain legible. Write and organize your work so that someone else could come into the laboratory and repeat the experiment using your directions without confusion and uncertainty. Completeness and legibility are key factors. **Make sure your notebook is signed by the TA at the end of each lab period.** (This does not mean that the TA has approved your method of taking data).

(D) Lab Reports:

The purpose of the laboratory report is to give a complete and concise description of the experiment. **All lab reports must be turned in to your TA one week after the experiment is completed.** NO LATE lab reports will be accepted by TAs. Unlike papers in other disciplines, lab reports are technical documents and should be written in a very specific way. Lab reports are not based on feelings or opinions, they are based on factual observations. Do not use the terms "I" or "we" in a lab report. Formal laboratory reports should be typed, with Times or Times Roman font (12 point), double-spaced, with 1" margins, and formatted according to the guidelines below:

—FORMAT—

- **Name**
- **Date**
- **Lab #: Title**

- **Abstract or Summary:** This is a condensed but thorough statement of the outcome(s) of the experiment. The abstract should be 2-3 sentences long, written in the past tense, passive voice.
  Example: Benzophenone was synthesized in 75% yield from benzene and benzoyl chloride using a Friedel-Crafts acylation. The product was a white crystalline solid (MP 100 °C) after recrystallization from 95% ethanol, and gave an IR spectrum consistent with an authentic sample.

- **Introduction:** This is the beginning of the body of your lab report. This section should give the reader a detailed background on the "what" and "why" of the experiment and put the experiment into a broader context. What was the purpose and/or objectives of the experiment? Why was the experiment conducted and why was it important in a broader context? (You should refer to your text book and other sources here). The introduction section should not simply state the obvious (for example, "This experiment was conducted in order to learn the technique of recrystallization.")

- **Materials and Methods (or Experimental):** This is a concise account of the materials and methods you used in the experiment. Since you will have already prepared a *detailed* procedure section for your notebook, you do not have to rewrite the procedures in your lab report. You should reference your lab text book (or hand-out) and describe anything you did that was different than the procedure in the book.
  Example: Thin layer chromatography (TLC) was used to separate spinach pigments according to the method of Gilbert and Martin (2001, section 6.2). Ethanol (95%), instead of dichloromethane, was used to extract the pigments from spinach leaves. TLC "spotters" (drawn from disposable glass pipets) were used instead of capillary tubes to apply the pigment solution to the TLC plate.
- **Results:** This is the core section of your lab report and before you begin writing it, you should gather together any and all data and/or observations you made during the

experiment. Numerical data are best represented in tables (or graphs when applicable) which should be labeled and put in the last part of your lab report (see below). State only your results in this section—the discussion section (see below) is the place to comment on the results and make conclusions; don't do that here. Your results should state only what you found in your experiment, not what you expected to find or what you were supposed to have observed. Be sure to write in the past tense, passive voice, no "I's" or "we's".
Example: The recrystallized benzoic acid appeared as a powdery, white solid weighing 0.67 g (54% recovery) and with a melting range of 98-101 °C.

- **Discussion:** Discuss your data and observations as they relate to the reactions and the experiment in a logical manner. You must talk about your results in a way that describes how your results support your lab objective. Exclude those points that are not relevant to your experiment. This is the part of the lab where you can explain or rationalize errant data or describe possible sources of error and how they may have affected the outcome of the experiment. Even if your experiment was a complete disaster you can still write an excellent lab report, as long as you understand what went wrong and can explain it, and the discussion section is where you can do that. In the end you should relate back to the introduction section and come to a definitive conclusion. Again, keep it in the past tense and scientific (no opinions, feelings, "I's" or "we's").

- **References/Literature Cited:** You are encouraged to use as many references in your in writing your lab report as possible, especially when writing the introduction where you're providing background and context. The internet is a valid source, as are texts. Books are cited using the format shown below:

  Lehman, J. W., *Multiscale Operational Organic Chemistry*, 1st Ed., 2002, Prentice Hall, *relevant page numbers*.

- **Figures, Tables, Data, Spectra:** The last part of your report contains the hard data—tables, graphs, spectra. All of these should be labeled and have a title, and you should include appropriate units.
  Example: Table 1. Simple Distillation of Acetone: Volume vs. Temperature
  Figure 3. Fractional Distillation of a Cyclohexane-Tolulene Mixture: Volume vs. Temperature
  Figure 4: IR Spectrum of 4-Bromonitrobenzene

- **Questions:** Questions from Lehman will be assigned for each lab report.

Note: Sample lab reports are posted on the web

Lab Schedule:

Week of	Lab #	Experiment # in Lehman	Procedure(s) to be completed
9/7	--	--	Safety Lecture and quiz
9/12	1	**1**: Learning Basic Operations (p. 20)	*Standard Scale* A. The Effect of pH on Sodium Benzoate B. Measuring the Density of an Unknown Liquid
9/19	2	**2**: Extraction and Evaporation (p. 28)	*Standard Scale* • Separation of Sucrose • Separation of Aspirin • Isolation of the Unknown Component
9/26	3	**3**: Recrystallization and Melting-Point Measurement (p. 35)	*Standard Scale* • Purification • Analysis
10/3	4	**4**: Heating Under Reflux (p. 40)	*Standard Scale* • Reaction • Separation • Purification and Analysis
10/10	5	**5**: Simple Distillation, Gas Chromatography (p. 49)	*Standard Scale* • Reaction • Separation • Purification and Analysis
10/17	6	**6**: Fractional Distillation (p. 58)	*Microscale* • Separation • Analysis
10/24	7	**12**: Vacuum Distillation, Optical Rotation (p. 101)	*Standard Scale* • Steam Distillation of Cloves • Isolation and Analysis of Clove Oil
10/31	8	**15**: Thin-Layer Chromatagrophic Analysis of Drug Components (p. 123)	*Microscale* • Purification of the Developing Chamber • Preparation and Development of the TLC Plate • Visualization and Analysis
11/7	No Lab		
11/14	Lab Final Exam		

Grades: Lab is worth 150 points (25% of your overall grade for the course), distributed as follows:

Component	Points
Safety Quiz	2
Lab Reports (8 @ 10 pts each)	80
Lab Technique	8
Lab Notebook	20
Lab Final Exam	40