



Chemistry 047 Laboratory

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Text: Ault, "Techniques and Experiments for Organic Chemistry" 6th Ed., University Science Books, 1998. Purchase or Rent from: University Science Books: <http://www.uscibooks.com/>

Grading: Quizzes (15%); Work Approach (5%); Safety Officers (5%); Lab report write-ups (75%)

There are no make-up lab sessions. If you miss a lab for a valid (i.e., medical or other true emergency) reason, you must provide your TA with a documented excuse for the absence. The laboratory grade will be based on your general ability to carry out the experiments, the accuracy with which you record and interpret your results, and your performance on laboratory quizzes. An example "lab report" will be handed out separately. **Note that messy or illegible lab reports will not be accepted. Items that are not legible will be marked as incorrect and will not be considered for re-grading.** Be careful with the carbon copy function of your lab notebooks.

Quizzes: Quizzes will be given prior to the start of laboratory work. They will cover material seen in previous lab sessions or material you should have learned as part of the prelab write-up. Chem. 047 lecture material may also be included on these quizzes.

Work Approach: Work approach points will be assigned by the TA. These points should be considered something you have to earn, not something that is taken away as a punitive measure for making mistakes. How do you earn these points? Approach your laboratory work in a safe, effective and efficient manner while being a good class citizen. The actual yield of your product will not be considered, but your approach to safety in the laboratory as well as your cleanliness and ability to apply good chemistry practices in your work certainly will. People who leave class early rather than taking time to help tidy up communal laboratory space will find it difficult to earn these points, as will people who are careless in their work habits, use poor chemical practices, show unsafe behavior or use flawed laboratory techniques.

Safety Officers: At the beginning of the semester students will sign up in pairs to be the "Safety Officers" for a given experiment. The safety officers for that day are responsible for leading an in-class discussion about potential safety issues, and waste handling guidelines for that experiment. These students will also be required to stay until the end of the laboratory to make sure the lab is tidy, that all glassware is clean, and that all equipment is put away.

Lab report write-ups: The majority of your grade will be based on your lab write-ups. These will be due one week from the completion of the lab work. **These reports must be handed in at the beginning of the next lab meeting.** If you do not arrive to lab with a completed report ready to be handed in at the **beginning** of the next lab meeting your report will be considered late and will lose 2 points. An additional 2 points will be docked each day the report is late. Note that each lab report is only worth 15 points total.

The lab report should contain the following sections:

Prelab: The T.A. will verify that a prelab has been satisfactorily completed before any lab work will begin. If the prelab is not satisfactorily completed it will be assigned a grade of zero for its portion of the overall grade for this lab and **the student will not begin lab work until the prelab is complete to the T.A.'s satisfaction.** At a minimum the prelab must include: the date; a statement about the purpose of the lab; a schematic drawing of the reaction to be done (or a drawing of the important chemical structures to be purified); a table showing the molecular weight, density (for liquids), number of moles, number of grams, and number of mL to be used for each substance used in the lab; *a list of potential hazards and precautions to be taken*; a drawing of important glassware to be used if it is a technique based lab; a brief experimental procedure written in your own words that you will follow.

Note: *The Sigma-Aldrich Chemical Company website (www.sigmaaldrich.com) is a huge repository of information about chemical substance including many physical constants.*

Observations and notes: This section must include a detailed description of what you actually did and what you observed. This should be in sufficient detail that anyone reading your notebook could repeat the process exactly as you did it, and understand exactly what you observed. Examples of standard notes and observations include: the time something was added, how long it took to add it, how long materials were allowed to react, gas evolution, color change, precipitate formation, reproduction of TLC results including *rf* calculations, tare weights, weights or volumes of materials used, etc.

Post-lab write-up: This section is where a significant portion of the grade lies. It should be a detailed discussion of the experiment including a discussion of the observations that you made, the results that you obtained, and an in depth description of the techniques you used. This discussion should show that you not only understand exactly what you did, but also should include a broader interpretation of what you learned through the experiment (i.e. why the technique you learned is important and/or an interpretation of your experimental results). Post-lab questions should also be answered in this section.

Academic Conduct: Cheating will be considered grounds for failing the course. All graded assignments must be your own work. Cases of cheating or plagiarism *will* lead to further disciplinary action which may include dismissal from the University according to the rules set forth in The University of Vermont's *Code of Academic Integrity*.

General Considerations:

Read the assigned reading (note that all experiments have reading assigned in the Ault textbook) and write a prelab *before* coming to class. The experimental procedure you will follow is either described in the text book, or in a supplemental handout, which is designated by "Handout" under Exp # in the following table. **Note: the experiments and order are subject to change depending on where we are in the lecture.**

Week of	Handout or Exp.#	Topic	Reading in Ault
Sept. 11	---	Introduction, Safety, Check in, Basic ChemDraw Tutorial	1-41
Sept. 18	Handout	Melting point and solventless aldol condensation (Lab Notebook, and MP)	44-45;150-158

Sept. 25	Handout	Solvent Recycling (Simple distillation)	62-78; 138-149
Oct. 2	Handout	Fractional distillation, GLC	70-72; 122-128
Oct. 16	Handout	Thin layer chromatography	116-119
Oct. 23	Handout and 28.2 (p.242) 29.11 (p. 261) 29.13 (p. 263)	Bromination of an alkene and qualitative organic analysis	239-241 247-248 266-272
Oct. 30	Handout	Extraction of carboxylic acid derivatives from neutral compounds	92-106
Nov. 6	Handout	Caffeine from Vivarin (recrystallization)	48-59; 317-318;
Nov. 13	Handout	Synthesis of 5,10,15,20- tetraphenylporphyrin and Column Chromatography	109-116
Nov. 27	E25 (pg 402-403)	Preparation of an <i>n</i> -alkylbromide	402-403
Dec. 4	E18 (pg 381-384)	Dehydration of methyl-cyclohexanol	381-384

Laboratory Experiment Abstracts:

Introduction, Safety, Check-in, ChemDraw Tutorial. Be sure to check your equipment carefully. Any missing or broken (cracked, chipped or otherwise in less than perfect shape) items should be replaced by the stockroom. Make sure your glassware is clean and dry before you begin your first experiment next week. A demonstration of how to use ChemDraw drawing software will be given.

Melting point and Solventless aldol. The purpose of this lab is to explore the technique of melting point determination and the effect of impurities on melting point. You will achieve this by determining the melting point of three solid samples that will be provided. You will also run a reaction that is an example of an “Aldol condensation reaction”; a reaction in which two molecules are combined into one product with extrusion of water. This particular reaction is unique in that no solvents are used. Solventless reactions are not always possible, but when it is possible to omit solvents it makes the reaction more environmentally friendly (“green”). You will collect the product of this reaction by filtration, dry it, determine the reaction yield and measure its melting point. **Give your sample to the TA for storage for use in the 7th experiment!**

Solvent Recycling: In this experiment you will be provided with dirty acetone (acetone is a common organic solvent used in many different cleaning applications) that must be purified so that we can use it throughout the rest of the semester as a cleaning agent. In this process you will learn the experimental

setup for simple distillation, which is a technique commonly used for the purification of liquid chemicals. Recycling this solvent through purification by distillation is an effective means of limiting the volume of hazardous waste that must be disposed of.

Fractional distillation, GLC. Fractional distillation is more effective than simple distillation at separating compounds that have similar boiling points. In this lab you will be assigned a mixture of liquids for separation by fractional distillation. Save 1 mL of this mixture for GLC analysis and fractionally distill the remainder using a stainless steel sponge-packed column. Be careful not to cut your hands on the stainless steel wire; it is sharp and very strong and should only be cut with scissors.

Thin layer chromatography (TLC). Here you will identify the components of common analgesics by comparing the retention factor (RF) of the components to the RF of standards. Silica gel TLC plates will be provided to you.

Extraction of carboxylic acid derivatives from neutral compounds. This experiment highlights extraction as an isolation and purification technique. It also emphasizes the concepts of pKa, solubility, and resonance.

Bromination of an alkene and qualitative organic analysis: This experiment highlights an alkene addition reaction. Bromine will be added to either E- or Z-stilbene to give a dibromide product. These stereoisomeric products will be compared by TLC and melting point analysis. In addition, three qualitative chemical tests will be performed to test for the incorporation of bromine in the product.

Caffeine from Vivarin. This lab introduces extraction and recrystallization as purification techniques. You will also recrystallize the product formed in the first experiment, determine the melting point of the purified material, and compare it to the melting point of the impure material.

Synthesis of 5,10,15,20-tetraphenylporphyrin. In this experiment you will synthesize 5,10,15,20-tetraphenylporphyrin from pyrrole and benzaldehyde. This reaction occurs in the gas phase at high temperature and thus is another example of a green (solventless) reaction. These reaction conditions are also green because they avoid the use of a strong acid promoter, which is typically required to effect this transformation under standard reaction conditions. Can you identify a non-green aspect to his experiment? You will purify the product by column chromatography, which is a standard and very important method of purification.

Preparation of an *n*-alkylbromide. Adapt the procedure in the book to prepare an alkyl bromide from the primary alcohol furnished to you.

Dehydration of methyl-cyclohexanol. Each student will dehydrate one of the methycyclohexanols (2-, 3, or 4-). The product obtained will be analyzed by GLC.

Laboratory Safety:

Organic laboratories are safe places to work if safety precautions are always observed. In general, the dangers are: cuts from broken glassware; the risk of fire when working with flammable chemicals; the risk of explosion when working with explosive chemicals, or closed systems; the risk of

exposure to hazardous (toxic or corrosive) substances. Caution, as well as careful thought and knowledge of the characteristics of what one is working with are necessary to avoid accidents and injuries. *Knowledge and preparedness are keys to safety.* If you have thought in advance of potential mishaps and solutions to those mishaps you will be prepared to deal with them in a calm and controlled manner. Minor accidents can snowball into major catastrophe due to panic.

Potentially hazardous apparatus and flammable, toxic, and/or corrosive chemicals will be used in this course. We will guide you in the safe handling and use of these materials, but ultimately your own actions will dictate your level of safety. The following rules and procedures must be observed at all times.

Rules:

1. You must wear safety goggles or OSHA approved glasses in the laboratory **at all times**. This rule will be strictly enforced. Do not wear contact lenses.
2. Know the location of exits, safety showers and eye-wash fountains.
3. Avoid personal contact with chemicals. Many chemicals have an adverse physiological effect (e.g. narcosis, toxicity, allergenicity, etc.). It is best to wear protective gloves. If you spill any chemical on your skin, wash it off at once with soap and water and tell your TA. Do not inhale chemicals or put them in your mouth.
4. Performance of unauthorized experiments is not allowed.
5. Horseplay in the laboratory is strictly forbidden.
6. Drinking, eating, or smoking in the laboratory is prohibited. All drink containers must be stored in your bag, or in the hallway. They may not be exposed in the laboratory.
7. Removal of chemicals and equipment from the laboratory is forbidden.
8. All accidents and injuries, however minor, must be reported to the instructor.
9. Extraneous sources of sound are not allowed.
10. Do not work in the laboratory while under the influence of drugs or alcohol.
11. Dress properly:
 - Do not wear open shoes, sandals, or crocks – **this will be strictly enforced!**
 - Do not wear baggy clothes.
 - Long hair must be tied back.
12. Do not pipette by mouth. (Yes, this used to be a thing).
13. When leaving the laboratory make sure all gas, air, water, steam, and electricity are turned off.
14. Protect your hands with gloves or a towel when pushing glass tubing or thermometers into stoppers or rubber tubing. Lubricate the hole to avoid glass breakage.
15. The working space, drawers, cabinet, and shelf above your bench should be neat and clean at all times.
16. You must clean up any spilled chemicals, this includes chemicals spilled on the balances or in the balance area.

17. Put broken glass in the broken glass disposal box; not in the trash.
18. Always point test tubes, flasks, and separatory funnels away from you *or other passers by*.
19. Carefully follow the instructions for proper waste disposal.

In case of accident

1. Fire. Personal safety is most important. Make sure everyone gets out of the room and the building. After the safety of all is assured, you may extinguish the fire. If a person's clothing catches fire, he or she needs help. Prevent the person from running. Put him or her under the safety shower and pull the chain. It is less effective to smother flames with a fire blanket. Never spray a person with a carbon dioxide fire extinguisher.
2. Chemicals. If corrosive chemicals are spilled on clothing, immediate showering with the clothes on is the best remedy. If chemicals are spilled on the skin, wash with large volumes of water. If a chemical is splashed in the eyes, wash immediately at the eye wash station. In all cases spills must be reported to the instructor.
3. Injuries. All injuries, no matter how minor must be treated immediately by a medical professional.

Housekeeping

The safety of a laboratory is directly related to the cleanliness of the laboratory. You will be held responsible for keeping this laboratory clean and un-cluttered. A part of your work approach grade will be determined by your housekeeping efforts. **The daily Safety Officers are responsible for making sure the lab is cleaned up, but they should not be expected to do all the cleaning! If you finish your experimentation early you will be expected to start the clean-up process.**

The following is a brief checklist:

- All equipment must be returned at the end of the lab period to whence it came.
- All glassware must be washed at the end of the lab period.
- All bench tops must be wiped down at the end of each lab period.
- All communal areas are the responsibility of the lab as a whole and must be cleaned and organized at the end of each lab.
- The chemical dispensing and waste areas must be kept clean and properly labeled.
- All gas, air, water, steam, and electricity must be turned off.

My signature below indicates that I have read, understood and will comply with the safety rules. I understand that my lab grade will be penalized and I may be dismissed from lab if I do not comply.

Signature: _____ Date: _____