



Chemistry 143 Laboratory Fall 2008

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Text: Ault, "Techniques and Experiments for Organic Chemistry" 6th Ed., University Science Books, 1998.

Grading: Quizzes (15%); Technique (5%); Lab report write-ups (80%)

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. 143 lecture material may also be included on these quizzes.

Technique: This grade will be based on several factors including (but not limited to) the quality of the chemical samples you hand in, your ability to conduct the experiment, your safety practices, and your housekeeping (i.e. the cleanliness of your work area when you leave).

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 20 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 brief experimental procedure written in your own words that you will follow.

Note: *If you do not have a copy of the Aldrich Catalogue (i.e. Aldrich Handbook) request one at: <http://www.sigmaldrich.com/Brands/Aldrich.html>*

This is an invaluable free resource

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. 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 and write up your prelab before coming to class. The experiments designated within each chapter describe the procedures that you will actually carry out in the laboratory. For several of the labs a supplemental handout will be given that more fully describes the procedure to be done; this is designated by "H." under Exp # in the following table:

Date	Handout or Exp.#	Topic	Reading
Sept. 9 th /11 th	---	Introduction, Safety, Check in	1-41
Sept. 16/18	Handout	Melting point and solventless aldol condensation (Lab Notebook, and MP)	44-45;150-158
Sept. 23/25	Handout	Solvent Recycling (Simple distillation)	62-78; 138-149
Sept 30 Oct. 2	Handout	Fractional distillation, GLC	70-72; 122-128
Oct. 7/9	Handout	Thin layer chromatography	116-119
Oct. 14/16	E.5 (pg 337-340)	Caffeine from Vivarin (recrystallization)	48-59; 317-318; 337-340
Oct. 21/23	E9	Eugenol from clove oil (extraction)	92-106, 346
Oct. 28/30	Handout	Liquid CO ₂ Extraction of Limonene	

Nov. 4/6	Handout	Synthesis of 5,10,15,20-tetraphenylporphyrin and Column Chromatography	109-116
Nov. 11/13	E25 (pg 402-403)	Preparation of an <i>n</i> -alkylbromide	402-403
Nov. 18/20	E28 (pg 407-409)	Nucleophilic competition	407-409
Dec. 2/4	E18 (pg 381-384)	Dehydration of methyl-cyclohexanol	381-384

Laboratory Experiment Abstracts:

Introduction, Safety, Check-in. Be sure to check your equipment carefully. Any broken or missing items which are cracked, chipped or otherwise in less than perfect shape should be replaced by the stockroom. You will be required to replace any missing or defective items at your expense at the end of the semester, so make sure you start with a well stocked drawer of unbroken equipment. Make sure your glassware is clean and dry before you begin your first experiment next week.

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 a "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 and determine its melting point as well. You will also determine the yield of the recovered material, bottle it, label it, and turn it in to your TA. It is critical that you give this material to your TA because it will be used in a later 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 sample 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. Here you will identify the components of common analgesics by comparing the RF of the components to the RF of standards. Silica gel plates will be available to you.

Caffeine from Vivarin. This lab introduces extraction as a purification technique. Experimental is noted in the text book.

Eugenol from clove oil. This lab further highlights extraction as an isolation and purification technique. Use 10 grams of clove oil (instead of 5) and methylene chloride as solvent. You will also learn vacuum distillation as a purification technique.

Liquid CO₂ Extraction of Limonene: The use of liquid CO₂ as an extraction solvent offers many safety and environmental benefits over standard organic solvents. For example, it is nonflammable, relatively nontoxic, readily available and environmentally benign. In industrial applications it is easy to recover the CO₂ (by controlling the pressure), thus making it possible to reuse the solvent many times with no net environmental impact. Liquid CO₂ has been used industrially for the extraction of essential oils, and in new greener methods of dry cleaning. In this laboratory we will generate liquid CO₂ and use it to extract limonene (an essential oil found in citrus fruits) from orange peel.

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. 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.

Nucleophilic competition. Carry out the experiment with the butyl alcohol assigned to you. Product analysis should be accomplished by GLC.

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:

The organic laboratory is a very safe place to work if safety precautions are always observed. 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 will be observed at all times.

Rules:

1. You must wear safety goggles or OSHA approved glasses in the laboratory. This rule will be strictly enforced at all times. 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.
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 or sandals.
 - Do not wear baggy clothes.
 - Long hair must be tied back.
12. Do not pipette by mouth.
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. The balances and balance area should be cleaned of any chemical spill.
17. Put 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 them off with large volumes of water. If the chemical is spilled in the eyes, it should be washed immediately at the eye wash fountain. 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 technique grade will be determined by your housekeeping efforts. 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: _____