

Chemistry 432 Laboratory Schedule

Additional Information

Fall 2014

Laboratory Textbook and Laboratory Supplies: The laboratory manual is available from Cal Copy. A highly recommended text, "The Organic Chem Lab Survival Manual: A Student's Guide to Techniques" by J. W. Zubrick is an additional useful source in providing details on laboratory techniques. **You must also have for this course: approved safety goggles (as specified in page 4), the yellow flame retardant apron, several pairs of nitrile gloves, a dish towel, 3x5" cards, rubber bands, a glass marking pen, soap and matches or a lighter.** An excellent cleaning soap for laboratory glassware can be made from a 1:1 mixture of Simple Green™ and Dawn™ detergent. Finally, you must wear appropriate personal protection equipment, goggles/glasses, apron and **non-open shoes** whenever any laboratory work is being conducted.

Wk # Date

1. 8/25 - 8/28 **Assignments:** Read material on safety, found in the inside front cover, review notebooks and format for preparative and investigative experiments, techniques pages 1-4, limiting reagents and theoretical yield, techniques page 5, and procedures to turn in products, syllabus page 4.
Discussion: The TA will first discuss laboratory safety then general techniques used in the laboratory course. Your TA will then prepare you for the theoretical and practical aspects of the methyl benzoate experiment. Course crashers will then be added to the class and everyone will check into laboratory lockers.
Lab Work: Check-in only.
Hand In: Check locker contents and sign inventory and safety sheets.
Last Day to Drop Classes: 2/4
2. 9/2 - 9/8 **Assignments:** Read the preparation of methyl benzoate preparation.
Discussion: **Quiz #1** General safety, the methyl benzoate experiment, limiting reagents and yield calculations.
Lab Work: Begin the preparation of methyl benzoate immediately after the completion of the quiz.
*****Note: Due to the conflict with Labor day the lab will start with Tuesday sections and end the following Monday.*** This pattern will continue throughout semester. ******
Last Day to Drop Classes: 9/4
3. 9/9 - 9/15 **Assignments:** Review the purification and isolation of methyl benzoate.
Discussion: Mechanism of the nitration of bromobenzene.
Lab Work: Complete the isolation of the methyl benzoate. Obtain an IR spectrum using the IR instrument in the CSL 502. Clean any necessary glassware used for the nitration experiment for the following week.
Hand In: Methyl benzoate.
4. 9/16 - 9/23 **Assignments:** Read the nitration of bromobenzene experiment, pages 8-11 and review the Solomons text on electrophilic aromatic substitution.

Discussion: Quiz #2 will cover the nitrobromobenzene reaction. Experimental details to the nitration of bromobenzene.

Lab Work: Begin laboratory work with the nitration of bromobenzene, when possible, work on earlier experiment. Before the end of this lab session, clean and wash glassware for the Grignard reaction so they may air-dry by the following week.

Hand In: Methyl benzoate report, including IR spectra.

5. 9/24 - 9/29 **Assignments:** Read preparation of triphenylmethanol experiment, pages 12-16. Phenylmagnesium bromide is prepared in the first part of this experiment then reacted with methyl benzoate to form triphenylmethanol. Preliminary write-ups for both of these reactions will be required. Assume that the Grignard reagent is prepared in 100% yield. Use 90 mmol of bromobenzene, 35 mmol of methyl benzoate and 85 mmol magnesium turnings.

Discussion: There will be a brief talk on laboratory safety and the Grignard reaction.

Lab Work: First, set up glassware for the Grignard experiment, adding 85 mmol magnesium to your round bottom flask (do not use an excess of magnesium, this will cause problems later in the work-up). Flame dry the apparatus starting from the bottom to top. Do not directly heat ground glass or teflon joints. Disassemble the apparatus as indicated in the lab manual and stopper with calcium chloride drying tubes in your locker until next week. Continue the isolation of 4-nitrobromobenzene.

Hand In: Methyl benzoate. 4-Nitrobromobenzene report including IR-spectra.

6. 9/30 - 10/6 **Assignments:** You must be well prepared for this laboratory. It is essential that you promptly begin experimental work.

Discussion: Final review of safety matters.

Lab Work: Time is critical, the class can not wait for you to complete your write-up. Re-flame dry your apparatus. After everyone has done this, only then will the ether be made available. **ETHER IS VERY HAZARDOUS TO USE IN THE PRESENCE OF SPARKS, FLAMES OR HOT PLATE HEATERS.** You need to get your Grignard reagent started as soon as possible and have the methyl benzoate added before the end of this laboratory period. While ether is being used in the laboratory no flames or other possible sources of ignition will be allowed. *Safety violations may result in your failure of this course.* Absolutely no visitors will be permitted during the experiment.

Hand In: 4-Nitrobromobenzene report including IR-spectra.

7. 10/7 – 10/13 **Assignments:** Be prepared to complete the preparation of triphenylmethanol and any earlier experiments.

Discussion: Quiz #3 will cover the Grignard reagent preparation and reaction with methyl benzoate as well as material on earlier experiments.

Lab Work: Complete the workup of the triphenylmethanol reaction. Distill off the diethyl ether using a heating mantel - **another flameless laboratory period.** Note: Sometimes the formation of crystals takes place over a period of weeks. There is no deadline for this experiment; turn in product as soon as it can be obtained.

8. 10/14 – 10/20 **Assignments:** Read 'The synthesis of Ethyl Cinnamate: An "on Water" Wittig Reaction', pages 17-20. Your laboratory notebook will be graded for this experiment based on the completeness and quality of your write-up. This laboratory period and

the next one are relatively easy and should be used to catch up on uncompleted experiments.

Discussion: The Wittig reaction, Green Chemistry, and silica gel chromatography

Lab Work: Begin 'The synthesis of Ethyl Cinnamate: An "on Water" Wittig Reaction' experiment

Hand In: 4-Nitrobromobenzene report.

9. 10/21 – 10/27 **Assignments:** Review material on the aqueous Wittig reaction.
Discussion: Quiz # 4 covering green and sustainable chemistry, as well as silica gel chromatography.
Lab Work: Purification and analysis of Ethyl Cinnamate.
10. 10/28 – 11/3 **Assignments:** Multistep Synthesis of Sulfanilamide – *Step 1. Synthesis of acetanilide.*
Discussion: Preparation of amides in organic chemistry. Electrophilic aromatic substitution. Mechanisms.
Lab Work: Conduct the synthesis of acetanilide. Dry the product for the next step. Take IR and melting points.
Hand In: All compounds should be turned in for grading by this time, except triphenylmethanol preparation and
11. 11/4 – 11/10 **Assignments:** *Step 2. Synthesis of p-acetamidobenzenesulfonylchloride.*
Discussion: Quiz #5 will cover formation of amides and electrophilic aromatic substitution. Please bring your calculator.
Lab Work: Conduct the synthesis of the sulfonyl chloride. Take IR and melting points. If time allows, conduct step 3.
Hand In:
12. 11/12-11/18 **Assignments:** *Step 3. Synthesis of p-acetamidobenzenesulfonylamide.*
Discussion: Continue with the multistep synthesis.
Lab Work: Conduct the synthesis of the sulfonyl amide.
*****Note: Due to the conflict with Veteran's day (Nov. 11) the lab will start with Wednesday sections and end the following Tuesday.*****
This pattern will continue throughout semester. ****
Hand In: Turn in all remaining products and the report on the synthesis of ethyl cinnamate!
13. 11/19-11/25 **Assignments:** *Step 4. Synthesis of Sulfanilamide (final step).*
Discussion: Quiz #6 will cover chemistry conducted in the multistep synthesis and infrared (IR) spectral analysis of compounds. Yield calculation, bring a calculator.
Lab Work: Continue and finalize the laboratory work, step 4.
Hand In: Turn in the final report by next week.
- 11/26-11/28 Thanksgiving break**
14. 12/1-12/4 **Assignments:** Final week
Discussion: Check-out.
Lab Work: Check-out. No laboratory work will be done during this final session, except cleaning of glassware and taking of melting points for unknown derivatives.
Hand In: Turn in the final report. Turn in all IR spectra of each intermediate and

final product, NMR spectra of intermediate made in step 3, and a sample of the pure final product. If time allows, indicate the efficiency of the antibiotic against bacteria (analysis conducted in a group format). Failure to turn in your final report with product (in a vial) will result in a minimal grade for this experiment.

You must carefully read and review the safety information found on the inside cover of this lab manual. Also, read the safety rules attached to your glassware inventory sheet, sign and return this sheet before you can begin any work in this course. There is **no compromise** regarding the safety rules. You must wear appropriate personal protective equipment whenever experimental work is being done. At the minimum these are goggles or safety glasses (ANSI Z87.1-2003 standard), full length lab apron and shoes or boots that completely cover your feet, or splash resistant booties. These items can be purchased from Grainger or the campus store. These items must be worn whenever any laboratory work is being done in the lab, whether done by you or anyone else. Visitors are not permitted in the lab. You are not allowed to bring or consume any food or drinks in the laboratory. Failure to follow these rules will cost you points and may result in expulsion from the laboratory. All chemical wastes must be disposed properly. Information regarding proper disposal of chemicals is found throughout the laboratory manual.

You must be prepared in advance for each laboratory meeting. You will not be allowed to begin experimental work prior to completion of pre-lab write ups or exercises. If you have not completed the pre-lab write up, you will not be allowed in the laboratory. Your laboratory notebook must be complete and up-to-date. You may use the same notebook you used for Chemistry 232. A preliminary write up is required, using the format shown on page 1-4 in the following techniques section of this manual. Preparative experiments must include tables, the correct limiting reagent, waste disposal and be initialed by your laboratory instructor before he/she will issue any chemical(s) from the storeroom. Take both your notebook and a **clean** screw cap bottle to the storeroom or from your TA to get your limiting reagent. We suggest that you record the weight of material received. Investigative experiments will use a slightly different format as given on page 4 of the techniques section. The pre-lab exercises need to be completed before coming to the laboratory. In most reactions you will turn in synthesized products in screw cap bottles. The bottle must be correctly labeled as follows:

Your name
Compound name
Weight of product in grams and % yield
BP or MP
Day and time of the lab section

and is to be submitted with a 3x5 card containing the following information:

Your name
Compound name
Weight of product in grams, theoretical and % yield calculations
BP or MP
Day and time of the lab section
Comments:

Solid products must also include two, (2), freshly filled melting point capillary tubes of your sample secured with a rubber band to the bottle. After the samples have been evaluated, the empty bottle and card will be returned to you. At the end of each laboratory session, room 502 will be cleaned up by the last two remaining students beyond the end of the normal class time. They will be responsible for disposing of paper wastes, standing water and etc.

Laboratory notebooks must be handwritten and should not contain loose notes or papers. Complete documentation of your observations is required. Neatness is nice but all observations should be made directly into your notebook. It is suggested that you write out what you intend to do prior to the beginning of the lab, indicating the important steps. As you do these steps, enter them directly into the notebook. If an error is made, place a single line through the error and write the correct information either above or behind it.

A large percentage of the points in the laboratory are devoted to the quizzes. It is imperative that you are well prepared for the six quizzes which will cover fundamental information on the experiments. **Quizzes are closed book, unless indicated otherwise.** The preparative experiments are graded on both purity and yield of material. If you can not turn in any product, but you have done the experiment you will be given a minimum score of 10 points. The Kinetic and Equilibrium experiment is graded on the basis of a logical write up. The multistep synthesis of sulfanilamide is based on a preliminary report, purity and product yields of intermediates (IR and NMR analysis) and final product, and is based on 40 points maximum score. You will also be evaluated (20 points) on your preparedness, attitude, proper disposal of chemicals and general safety.

Laboratory Grading Scale:

Six quizzes @ 20 pts each	120
Four preparative experiments @ 20 pts each	80
Multistep synthesis	40
Evaluation	<u>20</u>
Total points for laboratory	260