

Schedule of Experiments

Before coming to lab, download the experimental procedure and any other necessary handouts from the web, read that material and the assigned material from the text, do any OWL prelab assignment by the due date, and prepare a prelab outline. Downloads are found at " <http://copper.chem.umass.edu/> " - look under Undergraduate – Course Web Pages – Organic and Inorganic – Chem 268.

References given below are to your laboratory textbook, *Macroscale and Microscale Organic Experiments*, 5th Ed, by Williamson, Minard, and Masters. Bring to lab this text, your safety goggles, and your laboratory notebook, in which the prelab outline will be written. The required laboratory notebook is one in which a carbon copy of each page can be made and torn out. Before you may begin work, a carbon copy of the completed prelab outline and any other prelab material for that experiment must be submitted to your TA. Some references given below are to sections in Wade, which refers to the lecture text, *Organic Chemistry*, 5th or 6th Ed, by Wade. These refs provide background information.

Carefully read Chapters 1 and 2, and the handouts on Safety and Waste Disposal, Notebook and Grading Policies, and Make-up Policies and Procedures. You are responsible for knowing and following the contents of these handouts. Review and refer to this information throughout the semester. Refer to the text author's instructions on keeping a good notebook. This is given on the publisher's website – a link to this is given on the Chem 268 home page. (a second example is also posted on the Chem 268 website).

You must wear approved eye protection at all times while you are in the lab. Failure to do so will result in the loss of credit. Repeated failure to do so will result in expulsion from the course.

The weekly handouts for Chem 268H obtained from the web will provide changes and suggestions to experiments but the detailed procedure will be synthesized by you from the lab text and the handouts.

WEEK

- 1 Jan 31.** Check-in. EH&S Safety Training.
- 2 Feb 7.** Macroscale Nitration of Methyl Benzoate. Read Chapt 28. Review Recrystallization (Chapt4, especially macroscale recrystallization), MP (Chapt 3), and TLC (Chapt 8). Do Exp 2, p 433. Analyze crude and purified product by mp and tlc. Also analyze tlc of methanol filtrate from recrystallization. Analyze final product by ir. Read about making ir mulls – Williamson, pp. 244. Do molecular modeling handout. Ref: Wade, Chapt 17, especially 17-3, 17-7.

For extra help with SPARTAN, if needed, a tutorial from Chem 267H (Alkenes from Alcohols experiment) is available on the 267 website.
- 3 Feb 14.** Friedel-Crafts Acylation of Ferrocene: Acetylferrocene. Read Chapt 49, pp. 679-681, and Chapt 32. Do Microscale Procedure, pp. 473. Purify the product by column chromatography as follows: Column Chromatography. Read Chapt 9, follow procedures on pp. 210, 211 and p. 473, "Column Chromatography" and "Elution". (Basically to purify the acetylferrocene do Exp 4, p. 210, but use the acetylferrocene that you prepared, not a 50:50 mixture as described in Exp 4.) Analyze final product by ir as a mull.
- 3 Feb 21.** Grignard Synthesis - Macroscale. Read Chapt 38, pp. 528-532 and 539-543. Include the prelab exercise on p. 528 as part of your prelab outline. The apparatus will be modified slightly. Do Exp 4, 5, 7, pp. 539-543. The key word is DRY. Ref: Wade, 10-8, 10-9, 10-10, 18-12, and 14-2D.
- 4 Feb 28.** Grignard Synthesis (cont.). Finish WEEK 3. Reactions of Triphenylmethyl Carbocation. Use your product from the Grignard Synthesis. Read Chapt 33, pp. 477-481, 489. Do Exp 1 (micro), 2, 8. Ref: Wade, 6-13, 17-14C, 22-16.
- 5 Mar 6.** If necessary finish Reactions of Triphenylmethyl Carbocation. Wade: refer to appropriate chapters on IR and NMR of these types of compounds.
- 6 Mar 13.** Macroscale Esterification. Read Chapt 40, pp. 554-559, 563, 564. Review Extraction (Chapt 7). Read about macroscale extraction (Chapt 7, pp. 139-141) and macroscale distillation (Chapt 5). You will be assigned to synthesize one of the following esters: ethyl heptanoate, ethyl hexanoate, methyl heptanoate, methyl hexanoate, methyl octanoate, propyl hexanoate. BEFORE YOU COME TO LAB, using the procedure of Exp 4, pp.

563, 564, plan the synthesis and write out the procedure for the preparation of 0.080 mol of your assigned ester. Use an 8-fold mole excess of the alcohol (carboxylic acid is the limiting reagent). Obtain the physical properties (e.g., bp, sol, density) of the product and reactants from a handbook. Describe the odor of your product and of the reactants. Check the odor of different esters made by your colleagues. Analyze the product by IR and NMR spectroscopy and GC. Ref: Wade, 20-10, 21-2A. For IR, 12-8, 20-7A, 21-4A. For NMR, 20-7B, 21-4B.

- 7 **Mar 27.** If necessary, finish WEEK 6 and set up the Benzoin Condensation for **Week 8**. Over the week do Aromatic Resonance Effects in Cyclic Ketones. Ref: Wade, see Problem 16-15.
- 8 **Apr 3.** A Multi Step Organic Synthesis. Syntheses from Chapters 53, 55, 51, and chemical literature. Benzaldehyde to benzoin to benzil. Benzil to hydrobenzoin, tetraphenylcyclopentadienone, and benzilic acid.
- 9,10 **Apr 10, 17.** Multi-Step Organic Synthesis (cont.).

The following experiments follow procedures given at the publisher's website

<http://college.hmco.com/chemistry/organic/williamson/macroscale/5e/resources.html>

(look under "Qualitative Organic Analysis." Chapt 70 refers to the website)

- 11 **Apr 24.** Qualitative Organic Analysis. Read Chapt 70 (website), pp. 762-768, 769-771 (b, alcohols, c, aldehydes, and g, ketones), and 779-783 (2, alcohols, 3, aldehydes, and 9, ketones). Read Chapt 36 (lab text, omit Exp 4, 7). You will be assigned to identify an unknown compound (it may be an alcohol, aldehyde, or ketone). For the prelab outline, devise a plan to do so. Ref: Wade, 18-16, 22-3A,B, and for spectra, 12-8, 12-9, 18-5A,B.
- 12 **May 1.** Qual Organic Analysis - 2. Finish the identification of the first unknown. Devise a plan to identify a second unknown, which may be an alcohol, aldehyde, alkene, amide, amine, carboxylic acid, ester, or ketone. Refer to the appropriate sections of Chapt 36 (text) and 70 (website). Ref: Wade, review the appropriate sections on spectroscopic analysis and chemistry of the possible functional groups listed above.
- 13 **May 8.** Qual Organic Analysis - 2 (cont), Luminol, and Check-out. Finish identification of unknown 2. Do Luminol Exp. Finish and submit all remaining work. Check out and return key.

NOTE: If you do not check out properly, you will lose the credit equivalent to one experiment (10 pts).

All reports are due by Thur, May 15, NOON. REPORTS WILL NOT BE ACCEPTED AFTER THIS TIME.

Make-up Policy. To make up an experiment, a valid, well-documented excuse is required. All lab work must be made up within one week of the lab period which was missed. After this it will be considered to be late and will lose credit at the rate of one point per day. A TA signature is required on all make-up work. See the handouts, "Notebooks and Grading", and "Make-up Policies" for details.

Grades and Quizzes. See the handout, "Notebooks and Grading" for details.

Electronic communication. For your convenience, timely announcements about Chem 268H will be made via the Chem 268H web site and also through OWL. You may also contact the course instructor via email or in person.