

CHEMISTRY 2325 COURSE SYLLABUS

COURSE DESCRIPTION: Chemistry 2325 will develop techniques to further students' appreciation and understanding of modern organic chemistry. Emphasis will be on classical reactions, many of which received Nobel prizes. There is also a multi-step synthesis project.

PREREQUISITE: Chemistry 2315. Chem 2315 and 2325 **cannot** be taken simultaneously.

COREQUISITE: Chemistry 2320. Chem 2325 **cannot** be taken before Chem 2320.

INSTRUCTOR: Bub Carlson

Office hours: I am normally on campus from 8 – 4 and you are welcome anytime I am not in class. My class schedule is on my door. You can sign up for an appointment on my door or just stop by. TA's also have weekly office hours; see bulletin board and course website for details of TA office hours.

Room: 1340 HEB
Phone: please use e-mail
Email: mcarlson@chem.utah.edu

LECTURE: H (Sect 001) 11:50-12:40, HEB 2008
H (Sect 012) 3:40-4:30, HEB 2004
You may attend either lecture section, regardless of your lab section.

LAB: M (Sect 002) 1 – 5, HEB 4111 F (Sect 009) 1 – 5, HEB 4111
M (Sect 003) 5:30 - 9:30, HEB 4111 T (Sect 010) 1 - 5, HEB 4115
T (Sect 004) 1 - 5, HEB 4111 F (Sect 011) 1 – 5, HEB 4115
T (Sect 005) 5 - 9, HEB 4111 W (Sect 013) 1 - 5, HEB 4115
W (Sect 006) 1 - 5, HEB 4111 H (Sect 014) 5 – 9, HEB 4115
H (Sect 007) 1 – 5, HEB 4111 H (Sect 015) 1 – 5, HEB 4115
H (Sect 008) 5 – 9, HEB 4111 M (Sect 016) 1 – 5, HEB 4115
T (Sect 017) 5 – 9. HEB 4115

TEXTS: ***Organic Chemistry, Experiments and Techniques***
Authors: W.W. Epstein; Jimmy Lee Seidel; Craig S. Young
Published by Hayden-McNeil, ISBN 0-7380-1697-7 (new, revised edition)

Customized Lab Record, or similar bound notebook with carbon copies
Published by Hayden-McNeil, ISBN 1571829792

Lectures for Chemistry 2325 (copies of lecture transparencies),
Bub Carlson (optional, also on course website)

TEACHING ASSISTANTS: The laboratory teaching assistants have full responsibility and authority in the laboratory. Please respect their authority by being responsible individuals when a request is made.

Get your TA's e-mail address so that you can communicate with him/her in case you miss a lab or an assignment. **The TA's mailboxes are located in HEB 1504.** Be sure to put your TA's name on anything you put in the mailbox because all TAs share mailboxes. Always keep a computer copy of write-ups in case they are lost (and occasionally they are when they are not turned in directly to your TA).

STOCKROOM

ATTENDANTS:

The stockroom personnel have full responsibility and authority over laboratory and stockroom policies and procedures. Please respect their authority by being responsible individuals when a request is made.

RESPONSIBILITY:

You are responsible for all information and announcements given in class. If you miss a lecture or are late for a lecture be sure to get the information from another student. Please do not call and ask questions about this information.

ATTENDANCE:

Attendance is required. You learn by doing, not by the vicarious work of your friends. Because of limited availability of special equipment all students are encouraged to work in pairs on all experiments. It is recommended you switch lab partners frequently. Any student wishing to work alone may do so.

MAKE-UP LABS:

Make-up labs are permitted in certain circumstances. If you cannot attend your regular laboratory section for a legitimate reason, it is your responsibility to inform your professor to discuss the situation and get a signed permission form to do the make-up lab. You must pick up the form in HEB 1340; do not send an e-mail to inform your professor you must miss a lab, just get the make-up permission form. Give the permission form to the TA of the lab where the make-up work is to be done, and that TA will certify the work has been done and notify your regular TA. The make-up experiment should preferably be done that same week, or at the very latest the following week. No make-up labs can be done later than one week after the normally scheduled time.

POINT

BREAKDOWN:

1) Prelab	8 x 10	80
2) Lab work/notebook	8 x 40	320
3) Conclusions	8 x 50	400
4) Quizzes	8 x 20	160
TOTAL POINTS POSSIBLE		960

GRADING:

You are expected to complete eight labs. If you do not complete at least six labs you will automatically fail the course. Nine labs are scheduled, so if you miss one of the labs without making it up during the semester you can do the final make-up lab to substitute for the missed experiment if you have not already made it up. If you have done the first eight labs you can still choose to do the make-up lab to replace the grade of an earlier experiment.

Everyone who conscientiously does all eight labs with complete write-ups, and eight quizzes (earning a minimum of 50% of total possible quiz points) will receive an A or a B. Missing two or more labs will result in a fail. A missing write-up will be considered equivalent to missing half a lab. Five missing quizzes will be considered equivalent to missing one lab. Persons who fail to do all required work, but complete more than seven labs will receive a C or a D. Persons who do not take all quizzes, do not have a minimum of 50% of total possible quiz points, do not write full and conscientious conclusions, and do not turn in conclusions on time will not be guaranteed a minimum grade of B-.

For those who complete all the required work, at least half will receive an A and the rest a B. The A's will be at least half A and the rest A-. The B's will be at least one-third B+, about one-third B, and the rest B-. The percentage of points you earn will have no bearing on your grade since most of the points you receive are automatic for just doing the work.

For those who complete all the required work, you will be graded on a curve of A's and B's. TA's may vary slightly in their grading on quizzes and write-ups, so you will be graded only in comparison to other students in your TA's sections, not in any other TA's section.

QUIZZES:

Quizzes will be given at the end of each lecture covering the experiment done the previous week. Be sure to put your completed quiz in the correct folder for your section or your TA may not receive your quiz. One of the main purposes of the quizzes is to encourage you attend lectures. **Under no circumstances may quizzes may be taken early or late! Do not even ask!** There will be one make-up quiz given during the last class period over the make-up lab that can be used to replace a missing quiz or to replace the score of a lower quiz. You do not have to do the make up lab to take the make up quiz.

For the best performance on quizzes it is recommended you always do your lab write-up (or at least a draft of your write-up) before the quiz. Quizzes will emphasize lecture material.

Your quizzes and your write-ups should be returned to you by your TA by the following week so that you can learn from your mistakes. Keys to quizzes will be posted on the bulletin board outside of the labs. If your work is not returned by your TA within two weeks, notify your professor.

CHECK-IN:

Check-in will occur in the first laboratory period. You are required to bring a combination (not key) lock, goggles, and paper towels. Your lock combination number will be left with the attendant for emergencies. Failing to bring a lock after the second lab will automatically prevent you from doing any further lab work.

Many students register for the course and later decide to drop. Lab space is in high demand. Students who do not attend the first day of lab for check-in, or who are not on time will be assumed to be dropping the course and their lab benches will be given to someone else. Students who must miss the first lab for a reasonable excuse can reserve their lab bench by notifying your professor in advance.

If you switch sections, do NOT change your registration with the registrar. You instructor will change the section you are registered for. Changing sections with the registrar would require getting a permission code.

As part of the check-in you must be sure to sign the locker assignment sheet in the lab (with the correct number for the lab bench you have selected). If you do not sign the locker assignment sheet, then you are not registered for your lab bench and it may be given to other students registering late.

EQUIPMENT:

You are accountable for the equipment in your organic chemistry locker. On the first day of lab, your teaching assistant will assign a locker to you. Make sure to put a combination lock on the locker as you may be penalized for the replacement of missing items later during the quarter.

Located inside your equipment locker is a breakage card listing the entire contents of your locker. At check in, verify all items against the list. If anything is missing, the stockroom attendant will replace the item. During the semester, when you lose or break an item, the stockroom attendant will punch the card next to the item name to indicate that they have replaced the item. At the end of the semester, you may be assessed a grade point penalty based upon any breakage or loss in excess of that amount covered by your special course fee. Be careful with your equipment and do not lose the breakage card.

BREAKAGE:

Your laboratory fee is used primarily to cover the cost of chemicals and materials you use during the semester. It also includes a small component for small items in your drawer that are occasionally broken or lost. It does not cover breakage of special, major equipment that is not part of your equipment locker.

When doing the experiments part of your assignment is to work carefully without breaking equipment, particularly special equipment. If you do break a major piece of equipment during an experiment, you will lose all the points for that experiment (100 points). In most cases the loss of 100 points will cost you one or two grades (i.e., it would lower a grade of A to an A- or a B+). If you were to lose 100 points on three experiments, you would fail to complete the minimum number of required experiments (6) and you would fail the course.

As an alternative to losing 100 points, you may replace the equipment. You may obtain the equipment on your own, or the organic stockroom will replace it for you (at their wholesale cost). You can write a check, payable to the University of Utah Department of Chemistry, and make your payment at the organic stockroom. If you do not replace the equipment or write a check to replace the equipment by the end of the last week of the semester, you will automatically lose 100 points for the experiment in which the equipment was broken.

The costs for breakage of major equipment such as a 125 mL separatory funnel, a condenser, a 500 mL 3-neck round bottom flask, and a chromatography column are approximately \$65. Be careful when using this special equipment!

CHECK-OUT: Check-out will occur at the end of the last lab and generally takes about 15 minutes. You are required to clean all your glassware and review the glassware with your TA. Failure to check out will result in your grade being lowered one level (e.g., an A- to a B+).

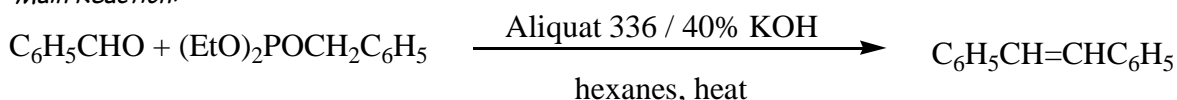
NOTEBOOK: The purpose of a laboratory notebook is to record all pertinent information about a laboratory procedure while the procedure is being done; this promotes the accurate communication of the procedure to the scientific community and/or any other interested party.

You are required to purchase the "Customized Lab Record" from the bookstore. This is a carbonless, duplicate set laboratory notebook that will allow you to give one copy of your notebook entry to the teaching assistant. You are required to get the signature of your TA on the bottom of the last page before leaving the lab for the day. You must follow the format presented below for your notebook entry. It is recommended you use the left side of the page for outlining procedures and the right side for observations. Read section 1.2 in your Techniques manual (the second half of the lab manual) for more information, including guidance in common calculations. You will submit notebook entries together with the discussion written at home a week after performing the experiment.

SAMPLE NOTEBOOK ENTRY:

A. Experiment #6: The Synthesis of Stilbene Using a Phase-Transfer Wittig Reaction

B. Main Reaction:



C. Physical Data (from: CRC Handbook 52nd Edition; Aldrich Catalog, 1988-1989):

	<u>PhCHO</u>	<u>(EtO)₂-POCH₂Ph</u>	<u>Aliquat 336</u>	<u>PhCH=CHPh</u>
molecular wt.	106.12	196.1	501.5	180.25
density	1.044	1.095	0.884	
b.p. (°C)	178.1	106.108 ^{1 torr}		305 ^{720 torr}
m.p. (°C)				124.5-124.8
ml used	0.25	0.55	0.25	
wt. used (g)	0.26	0.60	0.22	
mmol used	2.45 (limiting)	3.05 (excess)	0.44 (catalyst)	

D. Brief Procedure Outline:

Assemble a reflux apparatus with a 10 mL rb flask.
 Add 0.25 mL Aliquat 336, 0.25 mL benzaldehyde,
 0.55 mL diethylbenzylphosphonate, 2 mL
 hexanes, 2 mL 40% KOH.
 Reflux 1.5 hr.
 Cool 10 min, place in ice bath 10 min.
 Remove the lower layer.
 Collect crystals by suction filtration.
 Wash crystals: cold water x 2, cold ethanol x 2.
 Recrystallize from 3 mL ethanol and collect crystals.
 Take mp and determine yield.

E. Actual Procedure & Observations: (Brief procedure does not need to be repeated, just write actual procedure and observations on right side of page.)

Assemble a reflux apparatus with a 10 mL rb flask.
 Add 0.25 mL Aliquat 336, 0.25 mL benzaldehyde,
 0.55 mL diethylbenzylphosphonate, 2 mL
 hexanes, 2 mL 40% KOH.
 Reflux 1.5 hr.
 Cool 10 min, place in ice bath 10 min.
 Remove the lower layer.

Empty thermowell of sand, start magnetic stirrer,
 refill thermowell with sand.
 Add hexanes first—Aliquat 336 very viscous.
 Clean pipet (acetone, air) after each reagent.
 Wrap pipet with Parafilm so bulb fits.
 During reflux colorless reaction mixture turned
 A bright pink color.
 Cooling stops reaction. Crystals form.
 Use Pasteur pipet to remove lower (aq) layer.

Collect crystals by suction filtration.
Wash crystals; cold water x 2, cold EtOH x 2.
Recrystallize from 3 mL ethanol and collect crystals.

Use Hirsch funnel.
Use 1 mL each cold water, cold EtOH.
Needed about 4 mL hot EtOH to completely dissolve solid. Cool in ice bath. Crystals were white platelets.
Dry in oven for 10 min at 100° C.
Mp was 124.4-126.6°C. Yield was 0.21 g.

Take mp and determine yield.

$$\begin{array}{l} \text{Actual:} \quad \frac{0.21 \text{ g}}{180.25 \text{ g mol}^{-1}} \\ \text{Theoretical:} \quad \frac{0.25 \text{ mL} \times 1.095 \text{ g mL}^{-1}}{106.12 \text{ g mol}^{-1}} \end{array} = \frac{1.17 \text{ mmol} \times 100}{2.58 \text{ mmol}} = 45 \% \text{ yield}$$

NOTEBOOK:

1. Leave the first two pages of your notebook blank and make a table of contents.
2. Items A – D constitute the prelab. The pre-lab should be done before you come to the lab and will be graded by the T.A. Item E is to be done during the lab and F is to be done at home following the lab.) See example notebook entry after this section.
 - A. **Title.**
 - B. **Equations** (if applicable) **or molecular structures.**
 - C. **Physical data for all reactants used and products produced and solvents** (with literature reference cited) with appropriate precautions (when using dangerous chemicals such as sulfuric acid).
 - D. **Brief procedure outline.** Summarize the procedure, writing a sequence of well-defined steps that you are going to perform. Make sure that each step is described briefly so that you can easily read and follow it during the lab work. Avoid copying the text of the manual word for word. Write this outline on the left side of your notebook page.
 - E. **Actual procedure and observations (data sheets).** Include comments about what occurs during the experiment, like color changes, gas evolution, precipitates, etc. Make sure to write in such a manner that a person who would like to reproduce the experiment that you did can do so without getting verbal instructions from you and can then get identical results to yours. Be sure to write during the lab as you perform the experiment. Also include things that occurred that were not planned and which may or may not influence your results. Note that authentic description of the actual procedure sometimes demands recording the time. Write these observations on the right side of your notebook page.
 - F. **Discussion and Conclusion.** Explain your results and observations as well as any unexplained aspects of your experiment. Indicate if the goals were achieved. If they were not, do an error analysis.

3. At the end of the lab, you will tear out the carbon copy of your completed, signed notebook entry and hand it in to your T.A. before leaving.

The notebook entry is not to be a regurgitation of printed material, but a brief account of what you really did. In your conclusion, tie together the purpose with the results and offer explanations for low yield, etc.

PRE-LAB:

It is important that you have read the laboratory experiment in advance and are prepared to begin work. It is your responsibility to read the experimental procedure and background information until you understand the details of the

experiment. To assure your advance preparation you are required to submit a prelab sheet within the first 10 minutes of the lab or else no credit will be given for the prelab. This prelab will include sections A-D of the notebook record (see below). You may start work only after your TA has signed the prelab. No work is permitted without writing a prelab.

PHYSICAL
CONSTANTS:

Physical constants can be found in appendix 1 and 2 of your lab manual. They are also available at chemfinder.com on the web and in hard copies of the *Merck Index* and the Chemical Rubber Company *Handbook of Chemistry and Physics* in the Science Reserve desk on the fourth floor of the Marriott Library. To find the electronic edition of the *CRC* go to <http://www.lib.utah.edu> → research tools → article databases → C (or same route for the electronic edition of the Merck).

LAB WORK:

Record your observations on the right side of your notebook opposite the procedure you recorded for the pre-lab. Your lab work will be worth 40 points. Poor quality lab work and low yields cause a reduction of points awarded, especially for students who repeatedly achieve poor results.

CONCLUSIONS:

Your conclusions will be collected at the beginning of the next scheduled lab period. Conclusions 1 - 7 days overdue will be lowered 7 points for each day they are late. **Conclusions more than 7 days overdue will not be accepted.** Conclusions for the make-up lab will be due in your TA's mailbox (HEB 1504) one week after you complete the make-up lab.

Conclusions must be written individually, even if the experiment is done with a lab partner. A sample write-up is found on the web page.

The conclusions/final write-ups for the experiments should be sufficiently concise so as to not exceed two pages, but it is doubtful you can do an adequate job in much less than two pages. They should be done on a computer, double-spaced. A sample write-up can be found on the web page.

In Chem 2340 you will be conducting chemical reactions. It is important that your conclusions should **not** include procedure. They should concentrate on "why" and not "what". Your conclusions should be scientific and objective, and should not include any personal pronouns. Be sure to organize your conclusions.

You should begin the write-up by restating the chemical reaction and give its mechanism. The mechanism should never exceed ½ page. Explain **why** you did certain important things in the reaction procedure (why was a catalyst or a reflux necessary, why was it necessary to have anhydrous conditions, why was an extraction or recrystallization or cooling necessary, etc). What did some of your observations indicate about the reactions that were occurring? How were impurities, by-products, and unreacted starting materials removed during the work-up?

Give your percent yield and show how you did your calculation (indicating limiting reagent). Compare your melting point range with the literature value (and include a reference). Discuss the quantity and purity of your product, and do an error analysis on your results if you had problems. Explain the possible sources of a low yield or impurities and what you would do differently if you were to repeat the experiment.

You should emphasize concepts in your write-up that were emphasized in the lab lecture. Your TA will also give you an indication each week of important concepts

that you should include in your write-up.

If you use a reference for your conclusion, be sure to indicate it. Plagiarism in the conclusion is unethical and will result in a failure on the entire write-up.

For best results on quizzes it is recommended you always do your write-up (or at least a draft of your write-up) before the quiz. Flow sheets will be particularly helpful in your write-ups.

If you have difficulty writing coherent conclusions, you can go to the University Writing Center for help. They are located on the third floor of the Marriott Library. Their phone number is 587-9122 and their website is www.writingcenter.utah.edu/. If your conclusions are poorly written your TA will not accept them.

TECHNICAL REPORT:

A technical report on the two-week Sulfanilamide Experiment will replace the two conclusions for these labs and will be worth 100 points. It should be four pages in length, double-spaced typed, and follow ACS format. For an example of the ACS format see a *typical journal article* in Journal of Organic Chemistry on the course website. You can also find the ACS Style Guide on the web—go to University of Utah → Libraries → Reserve Readings → Reserve Catalogue → Chemistry 2340 or M. W. Carlson.

The technical report will not be any longer than two conclusions, but it will vary in style. It is necessary to cover both parts of the Sulfanilamide Experiment together because this allows consideration of the entire strategy of the multistep synthesis and the use of protecting groups. It is recommended a draft of the first half of the report be written before the quiz over Sulfanilamide I.

SPECTROSCOPY:

You are required to hand in a spectroscopy project by the last day of lab, the day of check-out. This project consists of a set of spectra—an infrared, an nmr, and a mass spec—all of the same compound. The set of spectra may be of any compound you have prepared during the semester, any starting material, or any intermediate in the multistep synthesis. You must identify the compound for the set of spectra and analyze the nmr and ir spectra. A sample analysis is given on the website. For the nmr spectrum indicate which protons give rise to each signal (aromatic signals need not be individually designated). For the ir spectrum indicate which functional group and type of absorption is responsible for each major peak (such as “alcohol O-H stretch”). For the mass spec analysis, determine the molecular weight of the compound. A sample analysis of an unknown is on the course website. The spectroscopy project is worth 20 points total, 10 points for identifying the compound and 10 points for analyzing the spectra.

DISABILITIES:

Any student needing special consideration because of a disability should contact the Center for Disability Services, 162 Union. It is recommended that any student who is pregnant delay taking organic laboratory courses until the pregnancy is complete.

The University of Utah seeks to provide equal access to its programs, services and activities for people with disabilities. If you will need accommodations in the class, reasonable prior notice needs to be given to the Center for Disability Services, 162 Olpin Union Building, 581-5020 (V/TDD). CDS will work with you and the instructor to make arrangements for accommodations.

All written information in this course can be made available in alternative format with prior notification to the Center for Disability Services.

LAB SCHEDULE FOR CHEMISTRY 2325

For online class materials for Chem 2325 see course listing under webct.utah.edu

Organic Chemistry Laboratory Spring, 2006

The order of the experiments is given below. They are not in the same order as they will be found in the lab manual.

Week beginning:	Lecture Topics (H)	Laboratory (M - F)
January 1/9	Introduction	No Lab
1/16	Lab # 1	Check-In (ML King Day Monday, Mon lab check in 1/23)
1/23	Lab # 2	Lab # 1 - Aromatic Nitration
1/30	Lab #3 Quiz over Lab #1	Lab #2 – Grignard Synthesis of Triphenylmethanol
February 2/6	Lab #4 Quiz over Lab #2	Lab #3 – Acylation of Ferrocene
2/13	Lab #5 Quiz over Lab #3	Lab #4 - The Wittig Reaction
2/20	IR Spectroscopy	No Lab (President's Day Monday)
2/27	Lab #6 Quiz over Lab #4	Lab #5 – Esterification: The Synthesis of Benzocaine
March 3/6	Lab #7 Quiz over Lab #5	Lab #6 – Reductive Amination
3/13	No Lecture (Spring Break)	No Lab
3/20	Lab #8 Quiz over Lab #6	Lab #7 – Sulfanilamide, Pt I
3/27	Make-up Lab Quiz over Lab #7	Lab #8 – Sulfanilamide, Pt II
April 4/3	Quiz over Lab #8 Quiz over Make-up Lab (optional; lab not required to take quiz)	Make-up Lab – The Aldol Condensation (optional), Technical Report due; Spectroscopy Project due; Check-out (required)
4/10	No lecture	No lab
4/17	No lecture	No lab