# Chemistry 333 (53): Organic Chemistry Lab Fall Semester -- 2013

Instructor: Dr. A. E. Moody	Office hours: in C3 (MG 3090) TBD
Office: MG 3140	in MG 3140 Tue: 1:30-3:30; more TBD AND by appt.
Phone: 785-4591	Recitation: 8:30 am TR, MG 2007
Email: amoody@truman.edu	Lab: 9:30 am to 12:20 pm TR, MG 1036

Chemistry is an experimental science. Thus, a chemistry lab is a central component of education in chemistry. Chemistry 333 is the two credit hour version of the organic chemistry lab at Truman, affectionately called the "Superlab". We will meet twice a week and, in a single semester, complete the same number and types of labs that would be done in both semesters of the two semester lab sequence. The lab work is intended to demonstrate the concepts and experiences of **both** Chemistry 329 and 331 lectures. (Thus, you **must** be currently enrolled in, or have successfully completed, Chem 331!) In addition, the experiments conducted will allow you to learn and practice fundamental lab techniques, e.g. crystallization and distillation, necessary to explore organic chemistry.

Each Tuesday and Thursday at 8:30 am, we will have a recitation lecture in MG 2007 to discuss the theory and technical details of the experiments. For safety sake, you must attend recitation! We will then immediately go to MG 1036 to perform the experiment. You will prepare for the experiment before these times by recording important information in your lab notebook. (See the instructions for keeping good lab notebooks later in this document.) It is also very important to "think through" the experiment before coming to lab so that lab time can be used effectively. The experiments are designed to be finished within the assigned lab period, which ends at 12:20 pm. Students cannot be allowed to work late on an experiment. Students who do not arrive on time, or do not use lab time efficiently due to inadequate preparation, demonstrate poor lab technique and will be penalized.

The course grade will be based primarily on your notebook, with additional points described in the table below. Lab notebooks will be checked in lab each day to insure proper pre-lab preparation. The lab notebook, lab reports, and products will be submitted for grading at times to be announced. The midterm exam is scheduled for Tuesday, October 8 during the recitation hour. The final exam will be given during the recitation hour on Tuesday, December 3. These tests will be based on the theory and techniques that we discuss and perform in the course this semester.

#### Approximate Grading Scheme

Safety Worksheet		20 points
Daily Notebook Preparation	~20 x 5 points each =	~100 points
Crystalline Product Quality (and Film Evaluation)	11 x 10 points each =	110 points
Overall lab notebook quality & instructor's observations of technique	·	50 points
Lab reports/notebook write-ups	18 x 25 points each =	450 points
Midterm and Final Exams	2 x 100 points each =	200 points
Total		~930 points

All lab work is potentially hazardous, but unsupervised lab work is even more dangerous. Students will not be allowed to work at times other than regular lab periods, even if the lab is open and in use by another group of students. Lab spaces are assigned so as to minimize crowding in work areas during each lab period. For that reason, students should come to lab only during their scheduled lab periods, unless they have been given specific prior approval for a change.

I take laboratory safety very seriously. No one will be allowed into the lab without protective eyewear. Clothing must be appropriate for the lab, or I will ask you to go change. No horseplay will be tolerated, with the penalty being ejection from the lab for the day and/or a zero on that experiment. (Further information dealing with safety is provided separately.)

# Tentative Schedule for Chem 333: FALL 2013

The text is **Making the Connections** by Padias; either first or second edition is OK. A <u>tentative</u> listing of the experiments to be carried out is tabulated below. Any changes made in the schedule will be announced in recitation or via email. The detailed experimental instructions are available via the Org-Lab website (http://chemlab.truman.edu/OrganicChemistry.asp.). You are responsible for downloading each experiment on

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	your own and	d in a tim	ely fashion!							
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Week of:		Tuesday	W	Thursday	F	S
Aug 18-24	Expt:			First Day of Classes! No lab! ;-)		
Aug 25-31	Expt:	Orient., Safety, check-in		Crystallization (Px2)		
	Text:	1-13		119-127, 20-24, 26-30, 37-43		
Sept 1-7	Expt:	Labor Day! No lab! ;-)		Solventless Aldol (P)		
	Text:			191-195		
Sept 8-14	Expt:	Melting Points		Fractional Distillation		
	Text:	49-53		141-152		
Sept 15-21	Expt:	Extraction: Acid/Neutral/Base		Continue Extraction (Px3, IR)		
	Text:	128-140		IR: 65-76		
Sept 22- 28	Expt:	Steam Dist'n: Eugenol (IR)	Int., Safety, check-inCrystallization (Px2) 119-127, 20-24, 26-30, 37-43a119-127, 20-24, 26-30, 37-43or Day! No lab! ;-)Solventless Aldol (P) 191-195ting PointsFractional Distillation 141-152action: Acid/Neutral/BaseContinue Extraction (Px3, IR) IR: 65-76-140IR: 65-76am Dist'n: Eugenol (IR) -155, 135Optical Activity & Biodiesel (IR) 56-60-155, 13556-60s-Alder (P, IR) + Qual. Spectra R: 77-101Catch upR: 77-101Fall Break: No lab! ;-)del-Crafts Acylation B2Ferrocene Chromatography (IR) 162-3, 172-7adi C=O UnknownMulti #2 Nitration (IR, NMR)in ard (P, IR, NMR)Finish Grignard; Qual Help 35in 41 [O] (IR, NMR)Luminol Synthesis (P)			
	Text:	First Day of Classes! No lab! ;-)Drient., Safety, check-inCrystallization (Px2)-13119-127, 20-24, 26-30, 37-43abor Day! No lab! ;-)Solventless Aldol (P)191-195191-195Melting PointsFractional Distillation9-53141-152Extraction: Acid/Neutral/BaseContinue Extraction (Px3, IR)28-140IR: 65-76Steam Dist'n: Eugenol (IR)Optical Activity & Biodiesel (IR)53-155, 13556-60Diels-Alder (P, IR) + Qual. SpectraCatch upIMR: 77-101Fall Break: No lab! ;-)Firdel-Crafts AcylationFerrocene Chromatography (IR)60-32Qual C=O UnknownQual C=O UnknownMulti #2 Nitration (IR, NMR)Qual C=O UnknownLuminol Synthesis (P)Aulti #3 Esterification (P, IR, NMR)Luminol Synthesis (P)Vidol (P) + ChemilumCatch UpTHANKSGIVING! No lab! ;-)THANKSGIVING! No lab! ;-)				
Sept 29-	Expt:	Diels-Alder (P, IR) + Qual. Spectra		Catch up		
Oct 5	Text:	NMR: 77-101				
Oct 6–12	Expt:	Midterm Exam		Fall Break: No lab! ;-)		
	Text:					
Oct 13-19	Expt:	Friedel-Crafts Acylation		Ferrocene Chromatography (IR)		
	Text:	30-32		162-3, 172-7		
Oct 20-26	Expt:	Multi #1 [O] (IR, NMR)		Qual C=O Unknown		
	Text:					
Oct 27–	Expt:	Qual C=O Unknown		Multi #2 Nitration (IR, NMR)		
Nov 2	Text:					
Nov 3-9	Expt:	Grignard (P, IR, NMR)		Finish Grignard; Qual Help		
	Text:	32-35				
Nov 10-16	Expt:	Multi #3 Esterification (P, IR, NMR)		Luminol Synthesis (P)		
	Text:					
Nov 17-22	Expt:	Aldol (P) + Chemilum		Catch Up		
	Text:					
Nov 24-30	Expt:	THANKSGIVING! No lab! ;-)		THANKSGIVING! No lab! ;-)		
	Text:					
Dec 1-7	Expt:	Final Exam				
	Text:					

You will check into a single lab desk drawer. The combination will be given to you; no other student will know it since these combinations are changed each semester. You are responsible for the glassware in your drawer. Replacement of lost or broken items will be charged to you. Prices for all items are on your check-in sheet and posted in lab.

Solid products are produced in many of our experiments and you will turn them in so that their quality can be evaluated. Your products should be labeled with the following: your name, my initials, their approximate due date, the name of the compound (the structure is not required), your measured melting point, <u>AND</u> the corresponding literature value. Labels are available in lab to remind to write each of these things on the sample.

### Notes about Learning and Grading

Labs present a chance to learn by doing, i.e. by working with the compounds and reactions that we regularly discuss in organic chemistry lectures. In this way, lab courses are an excellent opportunity to practice "active learning." I encourage you to discuss amongst yourselves (both before and during lab) the techniques and procedures that we will carry out in order to better understand them. Please ask questions of me (or the teaching assistants) if you don't see the point of a procedure. Discussion is a valuable tool to gaining understanding of this material.

However, <u>you must do your own labwork</u>, and when asked, come to your own conclusions. You will have some unknowns in the semester; it is your responsibility to determine their identity on your own. Upon completion of a lab in your notebook, you should sign and date it, indicating that it is your own work. The midterm and final exams are individual work; on these, a pledge must be signed that states that your work is your own. Signing the pledge will remind you of this obligation. In summary, learning together is highly encouraged, but the work that you turn in to be graded should be your own, reflecting your own understanding. I expect that each one of you will display this type of integrity in this course and in all of your work.

Late penalties may be assessed for labs not turned in on time. We will set a date together for each lab to be turned in, so that everyone has their lab experiment completed and enough time to finish the write-up. I will begin grading them at some random time after that due date. Until I begin grading, a late lab will have no late penalty. Once I begin grading, a late penalty of 10% is deducted from your score. If I complete the grading and hand the labs back, a late lab write-up will no longer be accepted, and your score for that lab write-up will be a zero.

<u>Accommodations of Students with Disabilities.</u> If you have a disability for which you are or may be requesting an accommodation, please contact the Disability Services office (x4478) as soon as possible, and let me know so that we can plan for it.

# The Laboratory Notebook

The notebook is the most important component in the evaluation of your work in this organic laboratory. You should read and follow faithfully the comments given below, as well as those given in your lab text (in Chapter 2). In addition, you may want to consult the library for further information about writing a laboratory notebook. (Note that those items in boldfaced print form a checklist for each experiment in your notebook.)

Your notebook must be <u>permanently bound</u> and <u>form "carbon" copies</u> of your writing -- I will grade the copies of each experiment as the semester proceeds so that you will always have access to your notebook to prepare for the labs. **Sign** and **date** each session of writing in the notebook. For your maximum learning, it is essential that you learn to acquire and use data independently. Therefore, you are NOT allowed to copy ANY information from notebooks of current or former students.

The pages of the notebook should be numbered and a table of contents at the front of the notebook should be continuously updated. Include a full reference to your textbook and our lab prompt web link on the table of contents page. Always write in ink, making sure that you write so that the copy page is legible and dark enough for my old eyes to read! Write only on the front of each page, leaving the backside of the page for calculations and scribbling. (Note that I will not be able to grade anything on the back of a permanently bound sheet!) The rule of thumb is that <u>if it cannot be easily read</u>, it is not adequately recorded in your notebook. I will not grade anything simply attached on loose leaf paper.

# Note that entries into the notebook should be written in three distinct stages:

## Stage 1: Before you come to lab:

- Read the chapters assigned and note carefully the experiment you are to do.
- Write the **title** of the experiment and the **date** that the work will be done in your notebook. Include a short statement indicating the **purpose(s)** of the experiment.
- If **chemical reactions** are involved, write overall reaction equations for them.
- Prepare a table of important physical properties and the hazardous properties of the ORGANIC materials (starting materials, solvents, and products) with which you will be working. Skim through the experimental section to be sure that your table includes all the organic chemicals that you will use. Always include the name of the compound, its molecular structure, and molecular weight. Other properties that may be important are melting points, boiling points, density, optical rotation, etc.... You are responsible for knowing the hazards of the substances that you use and how to handle them properly. This type of information is readily available online.
- Re-read the experimental section while you think through the manipulations. Write out the step-by-step procedure
  in your notebook (using the textbook to clarify the online lab prompt as needed), and leave space near each written
  step to write comments and changes as you carry out the procedure in the lab. [Many students like to make one
  column for these instructions (~2/3 of the width of the page) and another column beside the first for in-lab observations....] You may NOT use your textbook or the handouts from the web site in lab. All directions for the work that
  you do must be in your notebook. If you are to record data in tabular form in the lab, prepare a table for data ahead
  of time so as to save lab time.

## Stage 2: While you are in the laboratory:

Clarify and annotate the procedure that you follow, record the data you collect, and make observations. In the spaces beside the steps of your directions, *write your observations* and any changes that you make in the procedure. Write all data directly in the notebook, not on other paper. If you write something incorrectly, put a single line through the error, initial and date the line, then enter the correction. Do not obliterate any entry. It is a serious error to turn in at the end of the semester a notebook that has been rewritten or "copied over" outside of lab. Plan your work well so that all information is entered into the notebook properly while you are in the laboratory, not at some later time.

#### Stage 3: After you finish the experiment:

- Carefully include the details of the **mechanism** of any chemical reactions that you performed. Usually, your recitation notes are a valuable resource for this item.
- If you are carrying out a chemical reaction, calculate the theoretical and percent yields. (You can calculate a theoretical yield as soon as you have the precise amounts of reagents that you used in the reaction.) Sometimes, you will simply get back the same substance that you started with, so you compute percent recoveries. SHOW YOUR WORK on these calculations -- if I can see your errors, I can give partial credit!
- Draw graphs, calculate values, analyze spectra for important information, etc. as called for in the experiment. You'll need two copies of each of these items! Note that all graphs or other loose items to be included in the notebook should be <u>taped or glued</u> onto actual pages of your notebook, and their meaning should be described in words. Attach them so that they appear "upright" in the normal direction, and when you must fold an added item, please fold it only once. Do not have items hanging off of the notebook pages.
- Write a brief **summary statement or conclusion**. Briefly state the experimental results, what they mean, and explain what you could have done differently to improve your results. This statement should include whether you accomplished the purposes of the experiment.