

San José State University Department of Chemistry

CHEM 113B: Organic Chemistry Lab 2

Sections 3 & 4

Instructor: Dr. David Brook
Office location: SCI 166
Telephone: (408) 924-4994
Email: david.brook@sjsu.edu
Office hours: TuW 1330-1430 and by appointment
Class days/time: Lab: TuTh 1030-1320, Seminar Th 1330-1420
Classroom: SCI 139 (lab)
Prerequisites: Chem 113A (with grade of "C" or better; "C-" not acceptable), Chem 112B (may be taken concurrently with instructor's consent). Chemistry majors only or instructor consent.

Faculty Web Page

Copies of the course syllabus and major assignment sheets may be found on my faculty web page accessible through the quick links/faculty web page links on the SJSU home page after the first week of classes.

Messages related to this class will be sent by email. You are responsible for making sure I have an accurate email address for you that you check regularly. Emails to me should use my sjsu email address: *david.brook@sjsu.edu*, for questions, etc. Please put CHEM113B in the subject line of any emails related to this class

Course Description and Goals

From the Catalog:

Continuation of CHEM 113A including more advanced work. Prerequisite: CHEM 113A (or equivalent with grades of "C" or better; "C-" not accepted). Chemistry majors only or instructor consent. Pre/corequisite CHEM 112B

CHEM 113B is a continuation of 113A and includes more advanced techniques used in the synthesis, isolation purification and characterization of organic compounds. Emphasis is on the practical skills and knowledge required to execute multi-step syntheses and to design and/or adapt laboratory procedures rather than follow 'cook-book' procedures. Formal lab reports (following ACS/Journal of Organic Chemistry style) will emphasize writing and communication skills. Specific content is listed in the 'Schedule of experiments, Quizzes and Final'

Attendance of laboratory sessions (TuTh 1030-1320) and lectures (Th 1330-1420) is mandatory. Frequent absences from the lab will affect your progress in experiments and will

likely have an effect on your final grade. The lectures describe general background and application of spectroscopic techniques used in the class. In general they will not cover problem solving - you are expected to work problems on your own, including those from texts, problem sets and handouts.

Course Goals and Learning Objectives

Course Learning Outcomes (CLO)

- Mastering advanced laboratory techniques for manipulation of organic compounds (synthesis, separation, purification)
- Characterization of organic compounds by physical and spectroscopic methods including:
 - *Use of Infrared, 1D and 2D proton NMR and 13C NMR to characterize organic molecules*
 - *Apply mass spectroscopy (exact mass and fragmentation patterns) to organic structural analysis*
 - *Select conditions for GC analysis and analyze gas chromatographic data*
- Maintain useful contemporaneous notes of experimental procedures
- Write original formal laboratory reports in ACS journal style
- Locate scientific data as needed from paper and electronic sources as needed
- Design experimental procedures for new reactions and modify existing procedures as needed
- Operate safely in the laboratory and dispose of waste properly

Program Learning Outcomes (PLO)

Chemistry 113B satisfies the following Program Learning Outcomes for the Chemistry Department:

PLO #2 - Demonstrate understanding of core concepts and to effectively solve problems in organic chemistry.

PLO #6 - Answer questions regarding safe practices in the laboratory and chemical safety.

PLO #7 - Demonstrate safe laboratory skills (including proper handling of materials and chemical waste) for particular laboratory experiments.

PLO #10 - Write a formal scientific laboratory report, using the format and style of an article in a peer-reviewed American Chemical Society journal

Required Texts and Items

Texts

Note that exams are open book/note. Consequently electronic versions of texts are not acceptable since you **may not use cell phones or laptops during exams**

- Straus, Lab Notes for Chem 113B (purchase at SAACS in basement of Duncan Hall).
NOTE: you must purchase the current set of notes. Changes have been included this semester, including some relevant to safety. Use of notes from previous semesters will result in a fail for that experiment.

- Silverstein, R. M.; Webster, F. X.; Kiemle, D. J.; Spectrometric Identification of Organic Compounds, 7th ed. 2005
- Pavia, D.L.; Lampman, G.M.; Kriz, G.S. Introduction to Spectroscopy, 3rd ed (multicolor cover)
- American Chemical Society, the ACS style guide

Other equipment requirements

- Lab Notebook (that allows for duplicate pages)
- A set of molecular models.
- Pencils, Ruler, Calculator (Since you may not use cell phones or laptops on exams, you will need a calculator)

Lab Notebooks

The preliminary write-up of all notes and observations must be kept in a bound notebook with pre-numbered duplicate pages. All entries must be made in pen - NEVER erase or use 'white out'. Sometimes 'mistakes' turn out to be critical pieces of information! RECORD ALL OF YOUR NOTES AND OBSERVATIONS IN DIRECTLY IN YOUR NOTEBOOK, AS THEY OCCUR. Don't use scraps of paper; don't memorize measurements. The main point of the notebook is to be a journal of your laboratory activities so that you, or someone else, can read it at a later date and fully understand what you did, how you did it, and why things came out the way they did. It's OK to correct entries by drawing a line through them. The main point is that it is organized and understandable.

Lab Reports

All lab reports will be submitted online via canvas

The formal report must be in the style of a "Journal of Organic Chemistry" article - see the ACS style guide and any issue of JOC for details and examples. The report must be typewritten and double spaced and submitted on 'Canvas'. You need to include chemical structures (drawn with appropriate software, not hand drawn) within your report. Free chemical drawing software for windows can be found at <http://www.acdlabs.com> ("Chemsketch) and <http://www.mdli.com> ("Isisdraw"). Other packages include MarvinSketch and ChemDoodle. The chemistry department also has a (limited) site license for ChemDraw. (Ask me for details as to how to get a copy)

As with a published paper, the formal lab report should be written so that someone with a basic understanding of organic chemistry will be able to understand your experimental procedure, results and conclusions and be able to reproduce your experiment. Pay particular attention to the discussion points in the experimental handout provided.

Each report should also include, as an appendix, digital copies of all spectra recorded during the lab, along with digital copies of all relevant notebook pages.

Schedule of Experiments

Experiments and due dates are given in the "Schedule of Experiments" handout. Note that the lab reports are due online by the end of the day specified as the due date. A detailed prelab lecture about each experiment will be given on or before the start date of each experiment. In addition, supplemental lectures may be given as needed. The Schedule is subject to change and changes will be announced in class.

Preparation

A major requirement for success in Chem 113B is advanced preparation. This means you should read the experimental background and procedures carefully before the lab period. Since many of the experiments use more than one text, or multiple sections of a text, one suggestion is to photocopy the most important part of the procedures and have this as a ready reference (especially if only a few pages are involved).

You will be given the starting material for each experiment only after the instructor checks both your preliminary writeup in your notebook, and the table of reagents and products. Your starting material will be provided to you in a vial, which you will swap for an empty vial. If you need a second sample of starting material, you may be assessed a penalty. ***Note that you MUST have your preliminary writeup and table of reagents and products completed BEFORE you will be given any starting material.*** Obviously, if you have to work on these items during the scheduled lab period, you will seriously deprive yourself of adequate bench time to complete the experiment. A less obvious benefit of advanced planning will be that you may be able to use time during long procedures (e.g. refluxing) to perform other aspects of the experiment to catch up or get ahead. The lab schedule is arranged so there should be adequate time to complete each experiment well within the allotted dates.

Grading

The final course grade will be based on the total score of assignments over the semester:

5 experiments @ 10% each	50%
5 spectroscopy quizzes @ 4% each**	20%
Chemdraw assignment	4%
Final exam	26%
Total	100%

** A total of 6 spectroscopy quizzes will be given during the Tuesday 1:30 PM class periods (see schedule). The lowest score will be dropped.

No reports will be accepted after the final exam date.

Letter grades will be assigned to each lab report, the **total** score of the spectroscopy quizzes and the final exam. The final course grade will be a weighted average of these letter grades

The instructor may raise the final course grade by 0.33 grade point (eg., from B+ to A-) based on excellent preparation and laboratory work. A "bell curve" is not used to determine grades.

It is mandatory that all five Formal Reports be turned in (even if late) in order to receive a passing grade for CHEM 113B. All quizzes and the final exam are open to notes and textbooks used for CHEM 113B. Each student must have his/her own set (no sharing during quizzes and exams).

Attendance at all lab meetings will obviously be essential for you to complete the experiments. In general, work outside of the scheduled lab time will not be allowed except in unusual circumstances (e.g. medical absence, with verification). Any request for special circumstances must be accompanied with a verifiable document (e.g. doctor's note).

Equipment

You will be assigned an individual locker of equipment for your use during this course. You will be checked into your locker during the first lab period by the instructor, and sign an acknowledgment that you have all of your equipment. You are responsible for keeping track of all of the contents of your drawer. If you lose or break any item, you will be assessed a breakage fee at the end of the semester, so be careful with your equipment. It is possible to complete this course with a relatively small bill for expendable items: it is also possible to end up with a >\$100 bill! No one can help you if something is lost or broken, so be careful! When you check in, you will be given a coded check out pad from the storeroom. You may use this pad to check out additional equipment from the storeroom which may be required for a particular experiment. Note that certain equipment items checked out must be returned the same day to avoid a late fee. Remember, the code on your pad is assigned to you only, don't lose it, or someone else can use your code!

At the end of the semester, you must clean out your locker, replace all broken equipment or glassware, and have the instructor sign the check-out form. If this process is not completed fully, you may be charged a fee to clean and refurbish your locker.

Safety

Before you will be allowed to work in the laboratory, all students in Chem 113B must:

- a. Attend the safety lecture (first lab meeting) and
- b. View the Safety film.
- c. Read the Safety Rules for Teaching Laboratories on the chemistry department website
- d. Pass a short quiz on laboratory safety (grade of 80% or above)

All of the above conditions are mandatory, and must be completed before you will be allowed to work in the lab!

In addition to the points covered above, these are especially worth emphasizing:

- a. ALWAYS WEAR SAFETY GOGGLES IN THE LAB!!! (over your eyes, not on your forehead!)
- b. Be aware that we will be using some flammable solvents, do not have any flames when you (or someone else in the room) are handling these
- c. Similarly, treat all acids, bases, and reagents as potential hazards. Avoid skin contact with all of these, and treat any contact immediately. If you have a spill, never leave it unattended (let the instructor know).
- d. Glassware breaks. Use caution in any experimental procedure, and exchange any chipped or cracked glassware Also, dispose of all glassware in the special bins, not in the trash cans!
- e. The texts have sidebars or highlighted sections outlining special safety precautions - always enter these into your procedure section in your notebook (another reason to read ahead!)
- f. You are absolutely required to follow any instructions provided by the instructor related to procedures and/or safety. Failure to do so will result in your immediate disenrollment from this class.
- g. If you are not sure, ask!!

In addition, for safety reasons, before you start an experiment, you are expected to fully understand the procedures and hazards involved, and follow the instructors directions. Familiarize yourself with the safety rules for teaching labs on the chemistry department website. Note in particular: "Failure to comply with proper procedures and prescribed safety concerns shall subject the student to disciplinary action. 1) Any student who engages in unauthorized experimentation, or who seriously disregards safety, thereby endangering self or others shall be withdrawn immediately from the class with a grade of "F". 2) Any student who shows persistent disregard for safety may have his/her grade lowered, and may risk being withdrawn with a final grade of "F".

Special rules of safety and conduct apply when using the Bruker Fourier 300 NMR, the GCMS and other instruments. These will be provided by the instructor.

With preparation and organization, it will be possible to complete all of your lab work during the scheduled lab period. Except in unusual cases, no lab work will be permitted outside of the scheduled lab times. Usually, the only situation where this may be allowed is in cases of illness where more than a few periods are missed and for which you provide verification by your doctor. In any case, under NO circumstances are you to work outside of your appointed lab period without the written approval of the 113B instructor. Any student found working without expressed permission outside of the lab time may be disenrolled from the class!

Chemical Safety (CHEM 120S)

Chemistry 120S (Chemical Safety) is a required course for all chemistry majors and minors, and a prerequisite for all students involved in Chemistry 180 or 298 research courses.

Emergencies and Building Evacuations

If you hear a continuously sounding alarm, or are told to evacuate the building by an Emergency Coordinator, walk quickly to the nearest exit (out the door and turn left to exit the Science Building). Take your personal belongings as you may not be allowed to return. Follow the instructions of the Emergency Coordinators. Be quiet so you can hear instructions. Once outside, move away from the building. Do not return to the building unless the Police or the Emergency Coordinator announce that this is permissible.

Students planning to take CHEMISTRY 114

If you plan to take Chem 114 in the Fall of 2006 or later, note that the prerequisite of Chem 100W (or equivalent course approved by your advisor) will be strictly enforced. Students without a 100W course completed will not be allowed to enroll in Chem 114.

University Policies

Per University Policy S16-9, university-wide policy information relevant to all courses, such as academic integrity, accommodations, etc. will be available on Office of Graduate and Undergraduate Programs' [Syllabus Information web page](http://www.sjsu.edu/gup/syllabusinfo/) at <http://www.sjsu.edu/gup/syllabusinfo/>

Chemistry 113B Organic Chemistry Laboratory (Sec 3 & 4) Fall 2019

NOTE: this is a tentative schedule for the semester. Any changes will be announced in class in advance.

Course Schedule

Week	Date	Topics, Readings, Assignments, Deadlines
1	Aug 22	Introduction, greensheets, schedule, policies; safety lecture; check-in;
2	Aug 27, 29	Exp Zero
3	Sept 3, 5	Begin Exp One (stilbene) Chemdraw Assignment due
4	Sept 10, 12	Continue Exp One: Expt zero report due
5	Sept 17, 19	Continue Exp One Sept 19 @ 1:30 Spectroscopy Quiz One
6	Sept 24, 26	Finish Exp One, Begin Exp 2
7	Oct 1, 3	Continue Exp Two (Menthol), Expt One Report Due
8	Oct 8, 10	Continue Exp Two; Oct 10 @ 1:30 Spectroscopy Quiz Two
9	Oct 15, 17	Begin Exp Three
10	Oct 22, 24	Continue Exp Three, Expt Two Due Oct 24 @ 1:30 Spectroscopy Quiz Three
11	Oct 30, Nov 1	Continue Exp Three
12	Nov 5, 7	Begin Exp Four (dimedone) Nov 8 @ 1:30 Spectroscopy Quiz Four
13	Nov 12, 14	Continue Exp Four, Expt Three Due
14	Nov 19, 21	Continue Exp Four Nov 21 @ 1:30 Spectroscopy Quiz 5
15	Nov 26	Continue Exp Four (Nov 28 - Thanksgiving - No Class)
16	Dec 3, 5	Finish Exp 4, Checkout. Dec 5 @ 1:30 Spectroscopy Quiz 6
Final Exam	Dec 13	0945-1200 in Science 139 (or designated room); Formal Report for Exp 4 DUE (no reports accepted after the final is over).

Chemistry 113B Fall 2019 Lecture and Reading Schedule

DATE	TOPIC(S)	READING*
August 29	Intro to Structure Determination Review of Hydrogen Deficiency Index (HDI) Review Proton (¹ H) NMR and IR Introduce Carbon (¹³ C) NMR	PLK Ch 1 (all); Ch 2 (all-review); Ch 3 (all-review); Ch 4 (all) SWK Ch 2 (all); Ch 3 (all)
Sept. 5	Chemical Shift Interpretation and Prediction in ¹ H NMR and ¹³ C NMR	PLK Ch 3 (3.6; 3.10; 3.11); Ch 4 (4.2) SWK Ch 3 (3.4; App A; App B. 1; App B2a; App C; App D); Ch 4 (4.2.2; 4.3; 4.5; 4.7; App A-D)
Sept. 12	¹ H NMR coupling constants and splitting "trees" Use of HDI and Elemental Analysis; Rule of 13	PLK Ch 1 (all); Ch 3 (3.13-3.18); Ch 4 (4.13-4.16); Ch 5 (all) SWK Ch 3 (3.5; 3.12-3.14; App F)
Sept. 19	Spectroscopy Quiz 1	
Sept. 26	Isotopic vs Atomic Weights High Resolution Mass Spectroscopy (MS)	PLK Ch 8 (8.1-8.7) SWK Ch 1 (1.1 to 1.5 all sections)
Oct 3	Low Resolution MS; Elemental Analysis	PLK Ch 8 (8.1-8.7) SWK Ch 1 (1.5)
Oct. 10	Spectroscopy Quiz 2	
Oct. 17	2-Dimensional NMR	PLK Ch 10 (10.7) SWK Ch 5 (5.1-5.4)
Oct. 24	Spectroscopy Quiz 3	
Nov 1	Mass Spectroscopy Fragments I	PLK Ch 8 (8.8) SWK Ch 1 (1.5.4 to 1.6.17)
Nov. 7	Spectroscopy Quiz 4	
Nov. 14	Mass Spectroscopy Fragments II	PLK Ch 8 (8.8) SWK Ch 1 (1.5.4 to 1.6.17)
Nov 21	Spectroscopy Quiz 5	
Nov 28	Thanksgiving	
Dec. 5	Spectroscopy Quiz 6	

If time permits, additional topics may be included.

* **PLK** = Pavia, Lampman and Kriz, *Introduction to Spectroscopy*, 4th Ed, 2009

SWK = Silverstein, R.M.; Webster, F.X.; Kiemle, D.J., *Spectrometric Identification of Organic Compounds*, 7th ed, 2005

Problems in these books are not assigned, but you are encouraged to work on them to get additional practice! Also, ANY organic chemistry textbook will have basic spectroscopy questions for you to get more practice.