### CHEMISTRY 2123 LABORATORY (Amended!!)

Fall 2017

### 1 Credit Hour

### **Pre-Labs in CFS 103**

Sects: 11,12,13,14,15 Mon 1:00-1:50 PM Sects: 21,22,23,24,25 Mon 3:00-3:50 PM

### **Laboratories in CFS 119**

Sect 11: Tu 12:00-2:50 PM
Sect 12: We 12:00-2:50 PM
Sect 13: Th 12:00-2:50 PM
Sect 14: Fr 12:00-2:50 PM
Sect 15: Tu 9:00-11:50 AM
Sect 21: Tu 3:00-5:50 PM
Sect 22: We 3:00-5:50 PM
Sect 23: Th 3:00-5:50 PM
Sect 24: Fr 3:00-5:50 PM
Sect 25: M 6:00-9:50 PM

Instructor:	Dr. Benny E. Arney	Email:	CHM_BEA@SHSU.EDU
Office Phone:	294-1531 off-camp ext. 41531 on-camp	Office:	CFS 326 Or CFS 305 Or CFS 323
Website	Blackboard at www.shsu.edu	Office Hours:	Tu-We: 1:00-2:50 PM Th: 2:00-4:00 PM TuTh: 9:00-11:00 AM

# Lab is not "Show and tell". You are required to prepare, participate, perform and retain.

### **Prerequisites:**

Completion of CHM 1412 with a grade of C or higher and concurrent enrollment in CHM 2323 or prior completion of CHM 2323 with a grade of C or higher.

### **Required Texts:**

- (1) CHM 2123 Laboratory Manual,
- (2) "The Organic Chem Lab Survival Manual" by Zubrick (any edition) and
- (3) Your Organic Chemistry Lecture Text.

### **Required Supplies:**

- (1) Laboratory Research Notebook with duplicating pages.
- (2) DEPARTMENT APPROVED SAFETY GOGGLES
- (3) Black or blue non-erasable pen for writing in notebook.
- (4) A small container of a good grease-cutting dish-soap.

### **Suggested Supplies:**

- (1) neoprene gloves to protect hands.
- (2) a black "SHARPIE" marker to label your glass while in use.
- (3) Lab coat, to protect body and clothing.

Non-erasable pens (<u>BLUE or BLACK ONLY</u>) must be used for all laboratory work entries. Pencil or white-out are not acceptable and a minimum of 20% will be deducted from each laboratory for which they are used. Erasures will also cost an additional 20% of possible points for that experiment.

### **Important NOTICE!!!**

Organic Chemistry Lab requires attendance of a Pre-Lab on Monday afternoon:

Sect: 11, 12, 13, 14, 15 Monday 1:00-1:50 PM Sect: 21, 22, 23, 24, 25 Monday 3:00-3:50 PM

In this Pre-Lab session, the laboratory quiz will be given, and the laboratory preparation and instructions for the next experiment will be given. If you do not attend the Pre-lab for which you are scheduled, you will receive a zero for the lab quiz portion of the experiment.

# Cell Phones must be turn off during the Pre-Lab Lecture and the Laboratory and may not be used in the lab. If

your cell goes off in class, you <u>will</u> be ejected from the classroom. No ear mounted phones or ear phones (I-pods) are allowed in class. You are not allowed to use cell-phones or computers during laboratory. If you are caught using any electronic device (except calculator) during lab you will be ejected, your experiment shut down, and you receive a zero for that lab's grade.

### NO use of any electronic devices is allowed during lab without

the written documentation from the Office of Student with Disabilities.

### **Acceptable Attire:**

In the laboratory shorts, exposed mid-rifts, and open-toed shoes are unacceptable and will not be allowed. Long hair must be tied back and loose flowing clothing is highly discouraged. These are your safety and not for your convenience. We are thinking of your safety even if you are not. <u>Disfigurement and possibly death are not fashion statements you want to make</u>.

### **Attendance:**

Attendance is required at each scheduled laboratory Pre-lab and Lab session since each experiment will only be performed during the week it is scheduled and cannot be made-up. However, life is not always flexible so the lowest lab grade will be replaced by the percentage grade on the Laboratory Final. If you miss a lab session that will be the one replaced by the percentage grade on the Laboratory Final. Additional missed laboratory sessions will be recorded as zeros. **Late work will not be accepted** and the grade for that lab work will be a zero.

### **Check-In & Check-Out:**

At the first scheduled meeting of the in-laboratory class, each student will be assigned a lab cabinet and drawer containing equipment to perform the experiments scheduled for the semester. During this time make sure to examine each piece of glassware for chips, cracks, and breaks for your own safety and so that you will not be charged to replace it later. Make a note of any missing equipment on the check-in sheet provided in the Lab manual.

After you have "checked-in", you are responsible for the equipment in your cabinet, so do not leave it unlocked as one can rarely assume their neighbors are as conscientious as they are.

If you decide to drop the lab, resign from the University, or finish the course after you have checked-in then you must "check-out" with your TA. During "check-out" the cabinet is inventoried against the "check-in" sheet. If you do not "check-out", the TA will check-out the cabinet and you will be charged a \$25 Check-out Fee plus the cost of any missing or damaged items that were placed in your care.

### **Before Coming to Pre-Lab on Mondays:**

- 1. View and take notes from the PowerPoint for the experiment to be performed that week (if one is scheduled).
- 2. Read the relevant sections of the Zubrick book covering the techniques involved in the experiment (extraction, reflux, distillation, etc.).
- 3. Review the PowerPoint(s) for the previous Lab(s).

- 4. Have your Spectral Problems Assignment completed.
- 5. Review the material from the Spectral Problems.
- 6. Make note of questions/problems with the Lab experiment.

### In Pre-Lab:

Upcoming lab experiment will be discussed and outlined. Spectroscopy will be introduced and covered as part of every pre-lab lecture.

- 1. Spectral problem sets from the assigned sections of the lab manual will be taken up.
- 2. An overview of the week's experiment (if any) will been given.
- 3. An overview of the new assigned spectral problems will be given.
- 4. A quiz will be given that covers the previous experiment(s), the general details of the coming experiment, and the techniques used in the experiments, spectroscopy, and lab safety.

### **Preparation for the Lab**

Before coming to lab, you must prepare to perform the experiment, set up your lab notebook, and read and study any assigned or necessary materials.

- (1) Check schedule of experiments to find out which experiment is to be performed and any additional requirements.
- (2) Read the experimental description from the lab manual and any appropriate sections in the <u>Zubrick</u> book covering techniques to be used in the experiment.
- (3) Set up your lab notebook as described below. Experiment #1 does not need this step. **NOTE:** You will not be allowed to bring your lab manual to lab after Experiment #1. The only personal items allowed in the laboratory are your notebook, <u>Zubrick</u>, calculator, and blue/black nonerasable pen. If you bring your lap-top computer or other electronics to lab, you are responsible for them and their loss, damage, destruction, or theft are your responsibility alone.

### In Lab:

- (1) Upon entering the lab,
  - a. Turn in the laboratory notebook copy sheets from your ante-lab write-up (discussed below) and
  - b. The completed laboratory copy sheets of your Postlab write-up (discussed below) from the previous week's lab.
- (2) Come to lab, ready to work. There are only three hours available to work and no more. If you have not completed you work in the allotted time, the experiment will be shut down and points will be lost.
- (3) Perform your post-Lab write up in your notebook, see below.
- (4) Work safely.
- (5) As the experiment progresses, clean any used equipment and glass ware before putting it away in your drawers. Clean as you go, do not put it off until the end. **NEVER PUT DIRTY GLASSWARE AWAY.** Make sure all of your equipment is put away before leaving. Any equipment left out in the laboratory will be returned

to the stockroom for redistribution and you will be charged for any missing equipment at the end of the semester. 10% for each piece of glassware left out will be deducted from the grade for the lab experiment being conducted

## **Schedule of Experiments:**

Pre-Lab	Lab week	Experiment #	Pre-Lab Topic and Assignments	
Sept 11: Pre-lab Only			Overview of Lab requirements and Expectations, Begin Mass Spectrometry Sections 1&2. (Begin reading Chapter 16 in Karty)	
Sept 18:	Sept 18-22:	Building molecules: using molecular model kits, build 3D structures from the 2D depictions of Lewis, condensed, and lineangle formulas, observe rotation of single bonds, inversion of rings, chirality, etc.	<ol> <li>Check-in,</li> <li>Acid-Base Extraction and</li> <li>MS Section 3 and IR</li> <li>Section 1</li> <li>Turn in Spectral Problems</li> <li>MS 1&amp;2</li> <li>Turn Syllabi Signed Sheet.</li> </ol>	
Sept 25:	Sept 25-29:	Acid-Base Extraction Read Sections of Zubrick covering Extractions + Explores the use of acid-base properties of solutes in order to control water solubility by conversion to the charged conjugate acid or base. Used to separate a mixture of organic compounds.	1. Acid-Base Extraction and 2. IR Section 2 & 3 3. Turn in Spectral Problems MS 3 & IR 1 4. Begin reading Karty Chap 15.	
Oct 2:	Oct 2-6:	Recrystallization and melting point determination: Read Sections of Zubrick covering Recrystallization. Recrystallization as a purification method, selection of appropriate solvent, steps and techniques including fluted-paper filtration, vacuum filtration, melting point	<ol> <li>Recrystallization         Experiment     </li> <li>IR Sections 4 &amp; 5</li> <li>Turn in Spectral Problems         IR 2 &amp; 3     </li> </ol>	

		determination and percent recovery calculation.		
Oct 9:	Oct 9-13:	<b>Distillation:</b> Read Sections of Zubrick covering Distillation, theory, simple and fractional distillation	1. Distillation & & 2. Nuclear Magnetic Resonance (NMR) Sections 1 & 2 3. Turn in Spectral Problems IR 4 & 5	
Oct 16:	Oct 16-20:	Preparation of 2-Chloro-2-methylpropane: Preparation, characterization, and wet tests for alkyl halides.	1. Preparation of 2-Chloro-2-methylpropane 2. NMR Sections 3&4 3. Turn in Spectral Problems NMR 1 &2	
Oct 23:	Oct 23-27:	Dehydration of Cyclohexanol: Application of distillation to push a reaction to completion and purify the expected product. Tests for unsaturation (Baeyer test, Br <sub>2</sub> , Chromic Acid)	1. Dehydration of Cyclohexanol & 2. NMR Sections 5 & 6 3. Turn in Spectral Problems NMR 3 &4	
Oct 30:	Oct 30 – Nov 3:	Obtaining an NMR Spectrum Blackboard Handout: Each student will actually obtain an NMR spectrum of an unknown sample.	1. Obtaining an NMR Spectrum & 2. NMR Section 7 3. Turn in Spectral Problems NMR 5 & 6	
Nov 6:	Nov 6-10:	Preparation of an alkyl acetate + Read section of Zubrick covering reflux of reactions. Uses a simple reaction to introduce the use of the reflux technique and the application of extraction to the work-up of a reaction mixture to isolate the desired product.  1. Preparation of an A Acetate & 2. Spectroscopy Probl 3. Turn in Spectral Pro NMR 7.		
Nov 13:	Nov 13-17	Synthesis and Purification of Aspirin: Use of reflux to prepare a solid. Recrystallization and extraction for purification. Acidity of carboxylic acids and phenols and FeCl <sub>3</sub> test for	<ol> <li>Synthesis and Purification of Aspirin</li> <li>Spectroscopy problems.</li> <li>Turn in Spectral Problems.</li> </ol>	

		phenols	
Nov 27:	Nov 27:	Nov 27 - Dec 1:	Clean-up & Check-out, Written Lab Final during regular lab time

### **Notebooks:**

### Lab Prep Write-Up:

**Except for Experiment #1.** Prior to coming to the lab, the notebook must be prepared for conducting the experiment as outlined below. This is necessary, because you will not be allowed to bring the lab manual into the laboratory. You must perform the experiment using only your notebook and any additional instructions given in the pre-lab.

- **I.** First Page of Notebook with Name and Table of Contents.
  - a. Name and ID # at the Top.
  - b. Table of Contents to show |Page | Experiment Title
- **II.** For each Experiment
  - a. Title
  - b. A one or two sentence summary of the Experiment.
  - c. If a reaction is to be performed, the reaction should be shown using complete structural formulas.
  - d. Reagent table as follows: should include all reagents used in the experiment, MSDS sheets and chemical supply catalogs will available in the laboratory for gathering information needed. It is best to look up the information the week before.

Reagent	Formula	Mol. Wt.	mass or vol	mmoles	Cautions, Notes
sodium hydroxide	NaOH	40.0	5.0 g	125	Caustic, strong base
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e. Outline of experimental procedure with sufficient detail to actually perform the experiment. This is very important since the lab manual is not allowed in the laboratory.

### **PostLab Write-up:**

In the Laboratory

a. Describe your actual procedure. The true amounts of materials weighed out and equipment used. Include any observations such as temperature or color changes. For example:

"I weighed out 4.98 g of NaOH pellets and placed them into a 100 mL singleneck boiling flask. Two boiling chips were added to the flask with 25 mL of water. The dissolved NaOH was very warm, ...

- b. Calculation of percentage yield (if you don't remember how, find it in your freshman text, Chap 3 or 4).
- c. Discussion & Conclusion: Write your interpretation of the observations and results obtained during the laboratory. Why was the yield good or bad? Was the results what you expected? Et cetera.
- d. Answers to questions for the experiment.

**Grading**: For each Lab, a grade will be computed as follows:

Molecular models Lab: successfully checking-in and performing experiment 1 Attendance x (checkin and Experiment 1)

$$(0 \text{ or } 1)$$
 x 10

All Other Experiments:

Attendance x (Quiz(4pts) + Report(6pts)) = Grade  
(0 or 1) x 
$$(4+6)$$
 = max of 10

Spectroscopy Problem Sets: 10 points@.

The total of the labs make up 60% of lab grade.	40%
Spectroscopy Problem Sets	10%
Lab Practical	10%
Spectroscopy Final	10%
Lab Final (comprehensive) will count 30%	<u>30%</u>
	100%

 $A \ge 90\%$ ,  $B \ge 80\%$ ,  $C \ge 70\%$ ,  $D \ge 60\%$ ,  $F \le 59\%$ 

The report for each experiment will consist of the Laboratory notebook copy sheets of your labprep and post-lab write-ups with any additional questions from the experiment included in the notebook. The lab-pre write-up will be turned in at the beginning of the lab session for that experiment and the post-lab write-up will be turned after completion of the experiment at the beginning of the next lab experiment.

The lab practical will consist of demonstrating that the student can properly assemble lab apparatus, perform routine operations, perform simple calculations, and interpret routine lab tests.

The Spectroscopy final will consists of routine question over MS, IR, and NMR spectroscopy with particular emphasis on the relationship of molecular structure to the spectral data. Also there will be at least three spectral problems.

The final examination for the laboratory will be comprehensive and will focus on the experience of the laboratory. That is; how is this procedure done?; what should this result look like?; how do I set up this apparatus?; et al. Know your basic laboratory common sense. What were the tests performed in lab?, what reagents did they involve?, what were they testing for?, what was the reaction involved?, What does the test look like? What does a positive result for this or that test mean or look like? Fundamentals of spectroscopy? Remember to pay attention in the lab and record all of your observations in your notebook. Good-Luck.

Treat your laboratory experience like the first romantic date with the person with whom you plan to spend the rest of your life. Pay attention to what you are doing, why you are doing it, the chemistry involved, and not just worry about how soon can I get out of here.!! Focus on understanding what and why you are doing the procedures that you are performing and "what did they look like?".

Remember: If you do not know what you are going to do before you get to lab, you probably will not know what you did when you leave.

### !!!!! PREGNANT STUDENTS: !!!!!

The health and safety of your baby/fetus <u>cannot</u> be guaranteed. Exposure to vapors and solutions in the lab <u>will</u> occur. For most of the experiments, an alternative with less volatile, less toxic, or less noxious reagents simply does not exist. Since the purpose of the lab is to learn to perform common critical laboratory techniques, there is no dry alternative. So you have a choice of options:

- 1. Continue on with the lab experience unphased.
- 2. Obtain neoprene lab gloves and a self-contained respirator and then continue with the lab.
- 3. Take an X for this semester and complete the lab work in the next long semester for an official grade. This is only viable if the baby will be delivered before the next semester.
- 4. Drop the lab for this semester and take it later.

### STUDENTS WITH DISABILITIES POLICY:

It is the policy of Sam Houston State University that individuals otherwise qualified shall not be excluded, solely by reason of their disability, from participation in any academic program of the university. Further, they shall not be denied the benefits of these programs nor shall they be subjected to discrimination. Students with disabilities that might affect their academic performance should register with the Office of Services for Students with Disabilities located in the Lee Drain Annex (telephone 936-294-3512, TDD 936-294-3786, and e-mail <a href="mailto:disability@shsu.edu">disability@shsu.edu</a>). They should then make arrangements with their individual instructors so that appropriate strategies can be considered and helpful procedures can be developed to ensure that participation and achievement opportunities are not impaired.

SHSU adheres to all applicable federal, state, and local laws, regulations, and guidelines with respect to providing reasonable accommodations for students with disabilities. If you have a disability that may affect adversely your work in this class, then I encourage you to register with the SHSU Services for Students with Disabilities and to talk with me about how I can best help you. All disclosures of disabilities will be kept strictly confidential. NOTE: No accommodation can be made until you register with the Services for Students with Disabilities. For a complete listing of the university policy, see:

http://www.shsu.edu/dept/academic-affairs/documents/aps/students/811006.pdf

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