

CHEMISTRY 235
ORGANIC CHEMISTRY I LABORATORY

Corequisite: Chemistry 233: Organic Chemistry I

Spring 2013

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General Instructions:

1. Before any laboratory work is permitted you must read the WVU "Safety and Laboratory Rules for Organic Chemistry" and then sign a statement that you will abide by these rules. **Failure to abide by all safety and laboratory rules will result in a lower TA subjective grade and may result in (i) expulsion from the laboratory and/or Chem 235 laboratory class and/or (ii) assignment of zero for all experimental work during the laboratory period.**

NOTE: SAFETY GOGGLES AND LABORATORY APRONS ARE REQUIRED FOR ORGANIC LABORATORIES. They can be purchased at the University Bookstore. Covered shoes (not sandals or open toe/heel shoes) are to be worn at all times in the laboratory. Long pants or long skirts are to be worn instead of shorts.

2. Use a "Chemistry Spiral Bound Carbonless Copy Lab Notebook" (100 pg, Hayden-McNeil, ISBN #: 978-1-930882-74-4) for outlining your experiment. You must bring this notebook to each laboratory meeting. You must record all of your prelab plans, experimental observations and results, conclusions, and answers to experimental pre-lab and post-lab questions (found in the laboratory manual). Follow the guidelines in the manual (Chapter 2 and page 5) and on the attached sheets on how to keep a good laboratory notebook. Your teaching assistant will collect the carbonless copies and grade these weekly.
3. **Academic Dishonesty:** A full range of options is available to the instructor in the event of a discovery of academic dishonesty (i.e. cheating, copying results/observations/procedure/etc. from a previous/current student's laboratory notebook). These options are stated in the West Virginia University Student Conduct Code (see *the Student Conduct Code at http://studentlife.wvu.edu/office_of_student_conduct*), and include dismissal from school or assignment of an unforgivable "F" for a course grade. These options will be vigorously enforced.
4. Every student enrolled in Chem 235 must pay the laboratory fee (\$80) assessed by WVU. A registration restriction will be imposed if the fee is not paid. The laboratory fee is not refundable after the first week of classes. A charge for excessive breakage will also be levied.
5. Attendance is required. If you are forced to miss a laboratory period due to illness or an emergency, see your instructor. **There is no makeup lab for Chem 235!!** You are expected to attend ALL scheduled laboratory sessions.
6. If you withdraw from Chemistry 233, you must simultaneously withdraw from Chemistry 235 unless special permission is granted by the instructor. If you are thinking about withdrawing from Chemistry 233, please see your instructor.

7. A quiz will be given each week at the beginning of the laboratory period, so be on time. The quiz will be based on the experiment from the previous week and the experiment to be performed following the quiz. Study the experiments before you come to the laboratory! You are expected to understand the principles of the experiment and to know what you are going to do when you are in the laboratory.
8. Your teaching assistant (TA) is in charge of your laboratory section. Follow instructions made by your TA concerning lab safety, keeping the lab clean, experimental procedures, handing in assignments, etc. Do not be hesitant about asking your TA questions - he/she is there to help you. Failure to heed the instructions of your TA will result in a lower subjective grade. **Failure to abide by all safety and laboratory rules will result in a lower TA subjective grade and may result in (i) expulsion from the laboratory and/or Chem 235 laboratory class and/or (ii) assignment of zero for all experimental work during the laboratory period.**
9. **Grading:** The following grade distributions will be used to determine the grade that you receive in this course.

| Grading Category | Percentage |
|--|------------|
| Experimental Results: Unknowns | 10% |
| Laboratory Notebook (including preliminary write-up, questions, calculations, etc.) | 40% |
| Quizzes (lowest quiz grade automatically dropped) | 20% |
| Laboratory Final Exam | 20% |
| TA Subjective Grade (includes safety, following directions, attitude, neatness, housekeeping, preparedness) | 10% |
| Total | 100% |

Academic Integrity: *The integrity of the classes offered by any academic institution solidifies the foundation of its mission and cannot be sacrificed to expediency, ignorance, or blatant fraud. Therefore, I will enforce rigorous standards of academic integrity in all aspects and assignments of this course. For the detailed policy of West Virginia University regarding the definitions of acts considered to fall under academic dishonesty and possible ensuing sanctions, please see the Student Conduct Code at the Student Conduct Code at http://studentlife.wvu.edu/office_of_student_conduct. Should you have any questions about possibly improper research citations or references, or any other activity that may be interpreted as an attempt at academic dishonesty, please see me before the assignment is due to discuss the matter. [adopted 2-11-08]*

Academic dishonesty, as defined in Article III Section B of the WVU Student Conduct Code, will be dealt with according to University policy as described in Article IV.

Social Justice Statement: *West Virginia University is committed to social justice. I concur with that commitment and expect to maintain a positive learning environment based upon open communication, mutual respect, and non-discrimination. Our University does not discriminate on the basis of race, sex, age, disability, veteran's status, religion, sexual orientation, color or national origin. Any suggestions as to how to further such a positive and open environment in this class will be appreciated and given serious consideration.*

If you are a person with a disability and anticipate needing any type of accommodation in order to participate in this class, please advise me and make appropriate arrangements with the Office of Disability Services (293-6700).

Disclaimer: *The schedule, policies, and assignments within this syllabus are subject to change in the event of extenuating circumstances.*

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Text: Laboratory Manual for Organic Chemistry. A Microscale Approach, by W. R. Moore and A. Winston, McGraw-Hill, 1996.

| Week | Date | Experiment | Subject | Quiz |
|---------|---------|------------|---|-------------------------|
| Week 1 | Jan. 14 | | No Lab to Allow Schedules to Stabilize | |
| Week 2 | Jan. 21 | 3 | Check-in, General Instructions, Safety, and the Lab Notebook AND Melting Points . Also study Exp. 1 & 2. | Quiz 1 |
| Week 3 | Jan. 28 | 4 | Crystallization . Do both Macroscale (p. 22) and Microscale (p. 25). | Quiz 2 |
| Week 4 | Feb. 4 | 5 | Distillation . Students work in pairs and share data. One does simple distillation and the other does fractional distillation. | Quiz 3 |
| Week 5 | Feb. 11 | 6 7 | Gas Chromatography AND Thin-Layer Chromatography (do both GC and TLC) | Quiz 4 |
| Week 6 | Feb. 18 | 8 | Extraction: Separation of Unknown Mixture | Quiz 5 |
| Week 7 | Feb. 25 | 9 | Stereochemistry: Molecular Models . Each student should bring a set of molecular models to lab. You are advised to begin this exercise <u>before</u> coming to the lab. | No Quiz (worksheets) |
| Week 8 | Mar. 4 | 14 | S_N1 and S_N2 Reactions | Quiz 6 |
| Week 9 | Mar. 11 | 16 | E1 Elimination Reactions, Dehydrations of Butyl Alcohols | Quiz 7 |
| Week 10 | Mar. 18 | 19 20 | Stereospecific Bromination of <i>trans</i>-Stilbene (Procedure B.1 only) AND Diphenylacetylene(Procedure B.1 only) We will be doing a two-step synthesis of diphenylacetylene starting with stilbene. | Quiz 8 |
| Week 11 | Apr. 1 | 17 | Hydroboration-Oxidation of Alkenes | Quiz 9 |
| Week 12 | Apr. 8 | 18 | Reduction of Ketones to Alcohols with Sodium Borohydride | Quiz 10 |
| Week 13 | Apr. 15 | 10 11 | Infrared Spectroscopy AND Nuclear Magnetic Resonance Spectroscopy Study all three experiments: 10-12. We will work on assigning structures (Exp. 12) over the course of two periods. | Quiz 11 |
| Week 14 | Apr. 22 | 12 | Assignment of Structure by IR and ¹H-NMR Spectroscopy | Quiz 12 |
| Week 15 | Apr. 29 | | Lab Final Examination and Check-out | |

CHEMISTRY 235: LABORATORY NOTEBOOK
ORGANIC CHEMISTRY I
SPRING 2013

During the Chemistry 235 laboratory you must preliminarily outline (experiment title, date, introduction, theory) and report experimental procedures/data/results/conclusions for each experiment in a "Chemistry Spiral Bound Carbonless Copy Lab Notebook" (100 pg, Hayden-McNeil, ISBN #: 978-1-930882-74-4).

What is the purpose of a laboratory notebook?

The laboratory notebook allows confirmation/replication of experimental results and findings. If properly signed and dated, the notebook also establishes intellectual ownership and verifies first to invent. The laboratory notebook can be used as legal evidence in judicial proceedings and patent applications.

For Chem 235, you will use your laboratory notebook as a basis for your written laboratory report forms. The laboratory notebook is also worth 40% of your laboratory grade.

Who owns the laboratory notebook?

If you are carrying out research or laboratory testing procedures for a company (e.g. pharmaceutical company) or within academia, the corporation/institute that sponsored (financially or intellectually) or sanctioned your research/work has ownership of the laboratory notebook.

For Chem 235, you will be considered the intellectual stakeholder and will have ownership of your own laboratory notebook.

What should be included in the Chem 235 laboratory notebook?

Given below are some general guidelines of things to include in the laboratory notebook.

The ***Experiment Title and Date, Introduction, Theory, Table of Reagents, and Pre-lab Questions (Items 1-5 below)*** sections constitute your experimental outline and must be completed before you may begin work on the experiment.

The ***Experimental Procedure and Results, Data and Calculations, and Conclusion*** sections must be completed as you work through the experiment.

Post-lab Questions can be completed after the experiment or after you leave the laboratory.

1. **Title and Date:** include experiment title, date, numerical designation, pertinent literature references
2. **Introduction:** purpose of experiment or short statement of what will be covered/studied during the experiment and why. Also include conditions, apparatus, long term plans, preliminary calculations, safety concerns/considerations.

Should include ***"hypothesis-type"*** statements such as:....it is expected that...or...the results should show.....

3. **Theory:** chemical reactions with mechanisms, instrumental theory, calculations pertinent to the experiment.

4. **Table of Reagents:** tabular list of compounds that will be used with pertinent physical properties (e.g. molar masses, densities, melting point), preparations steps, and sample information.
5. **Pre-lab Questions**
6. **Experimental Procedure and Results:** Step-by-step written account of procedure recorded as you perform experiment. Record what YOU did during the experiment and not what the lab manual says to do. Record: weights of materials actually used, record YOUR raw data, experimental results, and observations, include (staple or glue) computer generated, charts, or tables of data, calibration information, repair, or maintenance information.

Will include “*experimental-type*” statements such as:....30.0 mL of liquid acetone was added to 1.256 mg of solid sample #1...or...it was found that the solution turned blue after 10 minutes.....
7. **Data and Calculations:** data collected during the lab (i.e., melting point, boiling point, refractive index of synthesized compound) and any calculations (i.e., theoretical yield, percent yield). Some researchers include data and calculations within the experimental procedure and results section.
8. **Conclusion:** a statement summing up the experiment, explaining data and why the experiment did or did not work for you, list possible sources of error and how such an error would affect the results, make suggestions for improving the procedure or your performance, include future plans (What would be the next step to continue the research?)
9. **Post-lab Questions**

What are some laboratory notebook DO's?

DO use a pen. Black pen is best.

DO include page numbers.

DO include a Table of Contents on the first 1-2 pages. (Leave the first 1-2 pages blank and update the Table of Contents throughout the semester.)

DO include the date, experiment title, and signature on each page.

DO sign and date each new entry. Best date: 12 May 2009 NOT 5/12/2009 which can be mistaken for 5 Dec. 2009 in Europe.

DO begin a new experiment on a blank page.

DO fix errors by drawing a single line through the incorrect entry and writing the correction above or below the incorrect entry.

DO initial and date the correction!!

DO include a description and/or drawing of apparatus used.

DO include computer generated graphs, IR spectra, etc. by stapling or gluing onto the appropriate page of the lab notebook.

DO include usual (temperature, ambient pressure, etc.) and unusual (power outage, hurricane, fire, etc.) laboratory conditions.

DO list your name on the outside and inside cover.

DO list some type of contact information on the inside cover in case of loss.

DO include the course title, semester date, section number, desk number, teaching assistant name (supervisor/project director in industry/academia), and room number on the inside cover.

DO use a bound, stitched, hard cover notebook with pagination or in our case a spiral bound carbonless copy notebook.

What are some laboratory notebook DON'TS?

DON'T use pencil.

DON'T erase or use white-out to delete entries.

DON'T scribble over or write over mistakes. Draw a single line through incorrect entry, write correction above/below, initial and date correction.

DON'T skip pages or leave pages blank. If a page or portion of page is left blank, place a large X on the entire blank area, initial and date.

DON'T rip out pages (except for the carbonless copy for grading purposes). DON'T record data on scraps of paper. Record all data in the laboratory notebook. DON'T record data/observations at a later time. Record as the experiment is completed.

Chemistry 235 Laboratory

Laboratory Fee: There is a laboratory fee of \$80 payable with your tuition. The laboratory fee is not refundable after the first week of classes. A charge for excessive breakage may also be levied. **IF YOU DROP THE COURSE, YOU MUST CHECK-OUT OF YOUR LABORATORY DESK.**

Laboratory Conduct: There is no smoking in the building. Each student is to do his/her own work in a quiet, efficient manner. The work area is to be kept clean and orderly. At the end of the laboratory period, the work area/sink should be clean and all common equipment should be returned to the common equipment containers. The gas jets should be checked to ensure that they have been turned off. The student should inspect the desk area to ensure that no apparatus has been left out.

Your teaching assistant will provide you with a tutorial at the beginning of each laboratory session. Students are expected to come to lab on time and listen and remain quiet until the tutorial is completed. Any questions should be directed to the teaching assistant at the end of the tutorial.

Laboratory Safety: Read the safety regulations carefully and adhere to them rigorously at all times for your own well-being and that of your fellow students. **YOU MUST WEAR SAFETY GOGGLES (NOT SUNGLASSES OR CONTACTS) WHENEVER YOU ARE IN THE LABORATORY.** **Covered shoes (not sandals or open toe/heel shoes) are to be worn at all times in the laboratory. Long pants or long skirts are to be worn instead of shorts.** Old jeans, a T-shirt and sneakers are the best laboratory attire. Portable radios, walkman, etc. are prohibited in the laboratory. If you must make/take a telephone call, exit the laboratory room. **NO FOOD OR DRINK IS PERMITTED IN THE LABORATORY.**

Laboratory Absences: Students should attend all laboratory sessions. Students arriving more than 5 minutes after the start of lab will not be permitted entry and will be given an unexcused absence and receive a zero for any laboratory unknown and/or assignments. Students absent because of severe illness or university business must speak with the instructor. There is **NO MAKEUP LAB** for Chem 235. **Since the laboratory experience is the chief component of the Chemistry 235 course, students absent from two or more scheduled laboratory periods will fail the course.**

Preliminary Work: Before coming to the laboratory, you should read through the experiment carefully. Make a list of all precautions, fill out the specified components in your laboratory notebook and plan the best way to do the experiment beforehand.

Laboratory Notebook: You must have and maintain a "Chemistry Spiral Bound Carbonless Copy Lab Notebook" (100 pg, Hayden-McNeil, ISBN #: 978-1-930882-74-4). You must bring this notebook to each laboratory meeting. You must record all of your experimental results/measurements, observations, and conclusions in this notebook. Before each laboratory session, write in your notebook an outline, which includes experiment title, date, introduction, theory, table of reagents, pre-lab questions, etc. Detailed instructions on how to keep an effective laboratory notebook are included herein.

Desk Locks: You will be given a combination lock for your desk. You are responsible for the equipment in your desk. It is your responsibility to open and lock your own desk.

Unknowns: Only one attempt will be allowed for each unknown. Check any calculations carefully (including significant figures and units) before you hand in your result. These results should be handed in at the end of the period. If you have not completed the experiment, notify your teaching assistant.

Special note: Students who are pregnant, or suspect that they may be pregnant, should inform the instructor and Ms. Barbara Foster, Safety Director, Clark 217, in person prior to attending the laboratory

Academic dishonesty, as defined in Article III Section B of the WVU Student Conduct Code will be dealt with according to University policy as described in Article IV.