

Chemistry 201 Laboratory

Summer, 2019

Instructor: William A. Price, Ph.D. Holroyd 345, *phone:* 215-951-1261; *email:* price@lasalle.edu

Required Materials:

Texts:

- (1) Laboratory Manual supplied by instructor.
- (2) Approved safety goggles with side protection. Prescription or reading glasses alone are not suitable substitutes. Safety goggles are designed to fit over such glasses. Also, because of the hazard associated with vapor irritation, the use of contact lenses in the lab is discouraged. Approved goggles can be purchased at the chemistry department stockroom for \$5.
- (3) A laboratory apron will be required at all times in the lab. Black rubberized aprons can be purchased at the chemistry department stockroom (cost \$10).
- (4) A bound laboratory notebook with numbered, copied pages will be required. This can also be purchased at the chemistry stockroom (cost \$15)
- (5) A pocket calculator with basic functions is recommended.
- (6) Optional: Rubber gloves that are rated solvent resistant (Nitrile gloves are provided)

Laboratory Objectives: The primary objective of the laboratory program for the Chemistry 201-202 sequence is to exemplify, through experimentation, the basic principles of Organic Chemistry. In actuality, the Chemistry 201 lab will emphasize traditional laboratory techniques suitable for a first course in Organic Chemistry. These techniques will include recrystallization, extraction, reflux, distillation and the determination of melting and boiling points. Each of the above may be done as a discrete operation, or in combination with other techniques. These basic techniques will form the foundation for performing later experiments primarily involving synthesis and structural analysis of organic compounds. In addition, instrumental techniques including Gas Chromatography (GC), Infrared Spectroscopy (IR) and Nuclear Magnetic Resonance Spectroscopy (NMR) will be introduced in the 201 course and expanded to include additional experiments in Chemistry 202. Since a major objective of the Organic Chemistry laboratory is to learn a large number of totally new laboratory techniques, you will be carefully observed on the safe and proper methods for implementing these techniques. You will also be evaluated as to the respect that you show delicate instruments, the safe handling and disposal of chemicals, the neatness of your work area and adherence to all safety regulations.

Safety: Please adhere strictly to all safety precautions as outlined by me and/or your lab text. Make yourself aware of the location of emergency equipment located in the lab, and know how to use each item. Consider every chemical that you handle to be, at the very least, toxic!

Read each label carefully. Nitrile gloves should be worn when dispensing chemicals. Be assured that violation of any safety regulation, especially the consistent failure to wear safety goggles **AT ALL TIMES** in the lab, may result in your immediate expulsion from the lab, and an assigned grade of zero for that experiment. Any chemistry faculty member may expel you from lab for not strictly following safety regulations.

You should wear practical clothing with little to no loose jewelry, tie your hair back and wear closed toe shoes – no sandals allowed!

Recitation: The first 20-30 minutes of each session will be devoted to a “recitation period”. Class will meet in its assigned recitation room to review any previous experiment, and to discuss the scheduled experiment for that day. Students are expected to be prepared for lab. If there are no student questions, the class will proceed immediately to lab.

Laboratory Notebook and Reports: Each student is required to have a sturdy, bound laboratory notebook with duplicate numbered pages. Upon obtaining your notebook, print your name, starting date, course and laboratory section on the front cover. Reserve the first page for a Table of Contents, and start each new experiment on a new page. The notebook will serve as an original record of everything that you observe during the experiment. **The duplicate pages are perforated and will serve as the laboratory report and the white pages as your copy.** Write in ink. The notebook should be organized in such a manner that another person reading it would be able to easily reproduce all of the steps that you performed in the experiment.

Before arriving at recitation each week you should have read the lab, completed the online quiz and prepared your lab report with the first 6 items as outlined in the standard rubric (see below).

All work in the lab is to be performed in pairs, unless otherwise instructed. The report is considered officially due during the next scheduled laboratory period following completion of the experiment. *20% of the maximum attainable grade will be deducted from the report for each week late.* No reports will be accepted after the last scheduled lab period.

A passing laboratory grade is necessary to pass Chemistry 201 and/or 202!

All lab reports must be turned in by 12 pm on the last day of classes to receive credit. **Reports are due one week after completion of the lab. If labs are late, 20% will be deducted the first week, 20% the second week late and reports will not be accepted 3 weeks after the date due.**

Academic Honesty: While you are encouraged to work with your lab partner, all lab reports are to be written and submitted in your OWN WORDS. This means that the calculations and discussions must be completed **individually** and not copied from any student or online source. The first incident of academic dishonesty will result in a grade of zero for that assignment. A second infraction could result in failure of the course. LaSalle University has an Academic Integrity Policy which can be found in the Canvas Help Menu under “Getting Help at La Salle”. You are expected to read and be familiar with this policy.

Your lab grade is determined by your performance during the lab period and based primarily on the quality and content of the lab reports that you submit. Lab quizzes are only available before the lab begins and cannot be made up afterwards. The technique grade is a subjective evaluation given by your instructor which considers your preparedness, punctuality, neatness, ability to follow directions, safety and overall lab practices.

Grade breakdown:

(1) Lab Reports	95%
(2) Lab Technique	5%

Final grade: (+/- will be noted)

A = 90-100%, B = 80-89%, C = 70-79%, D = 60-69%, F <60

Attendance: The laboratory schedule is set for the semester; as make-up labs are difficult to arrange, you are expected to attend every lab session. If you must miss a lab for a legitimate reason, you must let the instructor know **in advance**. Understand that it is **YOUR** responsibility to contact the instructor to make up a missed experiment. Labs are ideally made up the same week during an alternate lab section. This is only by special approval of BOTH instructors, and does not include labs missed because of studying for other classes or other non-emergency or non-illness reasons. Please note that each student case is unique and it is ultimately the instructor's decision to allow you to make up a lab. Because of the difficulty of arranging make-up labs, *a student may only make up one lab per semester as a rule*. Extenuating circumstances do occur from time to time, so please keep your instructor informed if this applies to you. Failure to notify your instructor of your absence BEFORE the lab begins will result in a zero for that lab.

Check-in: At the beginning of each semester, you and your lab partner will be assigned a locker. This locker will be used by only yourself and your partner. The locker will contain the common laboratory equipment including an **expensive** ground glass kit. On the first day of lab, you will be asked to check the contents of your locker and kit against a master list. Any missing or broken items may be replaced at no charge on the day of check-in only. After this day, missing or broken equipment can only be replaced by using cash at the stockroom. At the end of each lab, please make certain that you clean up all of the equipment that you used and return them to your locker. Lock the drawers and return the locker key to the board assigned to your section.

Students with Disabilities: Students with disabilities should refer to the student handbook for resources that are available to them as well as compliance with the American Disabilities Act. If you think you might qualify, please refer to in the Canvas Help Menu under "Getting Help at La Salle".

CHM201- Lab Rubric: the grading scheme for all laboratory reports is as follows.

Please record in your notebook **before coming to lab:**

- (1) Experiment number and Title
- (2) Your Name, lab partner's name, lab section
- (3) Date experiment is performed
- (4) **-2 pts Objective:** Brief statement of the purpose and topic of the experiment. What are the **new techniques** introduced by the experiment or what theory/chemical principle is explored.
- (5) **-5 pts** Intro or **Theoretical background**. A balanced chemical equation *with structures* should be shown or a brief description of the new technique. ****Be sure to cite references!**
- (6) **-5 pts** An organized **reagent table** listing the identity and physical properties of all reagents to be used (main reactants and solvents) including structure (for *organic* compounds), molecular weight, melting point (for solids) or boiling point (for liquids), density or specific gravity (for liquids) and concentration (where applicable). Also list particular safety hazards – i.e. *lacrymator*.

The following items should be recorded in your notebook **during lab:**

- (7) **-5 pts** An organized **data table** listing the identities and *actual amounts* of reagents used in grams or milliliters, and a listing of the number of **moles used** for all reactants.
(you may add this info to the existing reagent table if space allows)
- (8) **-5 pts** A **flow chart** outlining the experimental **procedure**.
- (9) **-5 pts** All **observations**, as they are made.
- (10) **-10 pts Results** – including grams isolated, observed melting or boiling points (distillation temperature), **theoretical yield and percent yield** should be tabulated. All calculations should be shown. Note: Product yield and purity may be included, where appropriate, as part of the lab report grade. Your **signature** at the bottom of each page, along with **my initials** as witness, should be completed before you leave lab.

Post lab write-up; other than the mechanism, please type this section:

- (11) A **Discussion** of the experiment paying particular attention to:
 - 10 pts:** an explanation of *why each procedure or technique was used*
 - 10 pts: Analysis of results:** Interpretation of collected data, yields, boiling points, melting points, etc. Conclusions drawn from each piece of data should be clearly explained. e.g. If the goal was to synthesize a specific product, explain how collected data supports that this product was created and comment on the purity of this product.
 - 10 pts: Sources of Error** – both human and non-human e.g.: “The observed 30% yield reflects a significant amount of unreacted starting material (name) as indicated by the spot seen by TLC.” Explain possible side reactions or competing reaction (what happened if water was mixed with reagents)
 - 3 pts:** Include **references** for all literature properties (*Aldrich catalogue, Chemfinder®*)
- (12) **-5 pts** A detailed **mechanism** showing the reaction YOU performed with your particular reactants. Arrows must be drawn correctly and the reaction should be shown from beginning to end, including the quench if that was a necessary step in product formation.
- (13) **-15 pts Supplementary questions** for each experiment will be assigned at the end of each recitation period. These should be answered, and included as the very last part of the laboratory report.
- (14) **-5 pts Conclusion:** One sentence – was the objective accomplished? How do you know?
- (15) **-5 pts Overall quality** of lab including preparedness, techniques and results.

The Laboratory Notebook

Record keeping and data interpretation are skills which you will use throughout your scientific career. The ability to prepare an adequate and reliable record of results is a fundamental requirement for all successful experimental work. One of the most important skills acquired during this laboratory period is how to accurately record your experimental procedure, observations and results. For this reason, the

Record must be able to be understood by 2nd person

The most important criterion for an acceptable lab notebook is that the record be complete enough for a second person to be able to follow your experimental work, thus being able to repeat the experiment and to obtain the same results. The record must be written while you do your work. Your records should reflect your work so well that any odd results can be interpreted later, even if you don't notice them at the time. Later, you can do calculations, etc.

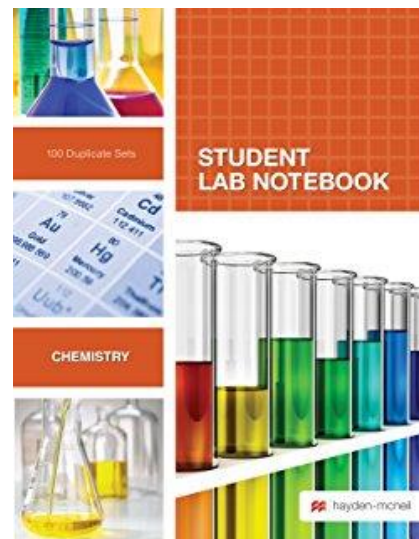
Lab notebook must be permanent record

Permanent entries: All entries in the laboratory notebook are to be made in ink. If errors are made, simply cross them out with a single line. Never obliterate entries; at a later time you may find that this information is useful. All data and observations are to be entered directly into the notebook, not on scraps of paper.

Permanent binding: Similar to the requirement that entries be made in ink is the requirement that the lab record should be kept in a book with a permanent binding. Students should purchase a bound lab notebook that makes duplicate copies. These are available in the chemistry stockroom. Each week, one copy will be submitted with your lab report and the other is your permanent record. The lab notebook will be signed each week by both you and your lab instructor. It is important that students begin to develop the skills needed to maintain the integrity of the record of their experimental work.

Entries must be timely

While you may want to wait to record data until after the lab is over in order to have a neat notebook it is more important to have a complete, accurate record of all original data and observations, complete with smudges, spills, and cross-outs. Your laboratory notebook should provide a complete record of the work which you have done through the year in the lab. If it is necessary to omit information on a page in your lab notebook, place a large X over the information but do not remove the page from the notebook. (Pages should never be removed from a lab notebook.)



CHL 201- Laboratory for Organic Chemistry I
Summer Session I, 2019

Instructor: William A. Price, Ph.D.

Location: Holroyd Hall 305

Texts: Custom Lab Manual by Mohrig, Hammond, Schatz, and Morrill
From *Modern Projects and Experiments in Organic Chemistry*

<u>Date</u>	<u>Experiment</u>
May 20	Introduction, Check-in, Safety
May 22	EXP 1 , Analgesics and Synthesis of Aspirin
May 23	EXP 2 , Extraction and Evaporation: Separation of the Components of "Panacetin".
May 29	EXP 2 , Identification of Unknown Component and Recrystallization
May 30	EXP 3 , Hydrolysis of an Unknown Ester: Hydrolysis and Azeotropic Distillation
June 3	EXP 3 , Extraction and Recrystallization
June 5	EXP 3 , Distillation, Boiling Points, Melting Points, Identification
June 6	EXP 4 , Isolation of (R)-(+)-Limonene from Orange Peels
June 10	EXP 4 , continued.
June 12	EXP 5 , Radical Chlorination Reactions
June 13	EXP 6 , Nucleophilic Substitution Reactions
June 17	EXP 7 , Acid-Catalyzed Dehydration of 3-Methylcyclohexanol
June 19	EXP 8 , Free-Radical vs. Ionic Addition of HBr to Alkenes