Chemistry 202 Laboratory
Spring, 2016

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Required Materials:

(1) Laboratory Text: Package containing *Techniques in Organic Chemistry*, third edition (2010), together with custom laboratory manual from *Modern Projects and Experiments in Organic Chemistry,* second edition, J. R. Mohrig, C. N. Hammond, P.F Schatz, and T. C. Morrill W. H. Freeman, 2003.

(2) Approved safety goggles with side protection. Prescription or reading glasses alone are not suitable substitutes. Safety goggles are normally 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.

(3) A laboratory apron will be required at all times in the lab. Black rubberized aprons can be purchased at the chemistry department stockroom.

(4) Rubber gloves that are rated solvent resistant.

(5) A bound laboratory notebook with numbered pages will be required. This can also be purchased at the stockroom.

(6) A pocket calculator with basic functions is recommended.

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 202 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 wore 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 *closed toe shoes – no sandals allowed!*

Recitation:

The first 20-30 minutes of each session will be devoted to a "recitation period". Students are expected to be prepared for lab. Prior to recitation, each student is complete a short lab quiz that can be found on Canvas®. Class will meet in its assigned recitation room to review any previous experiment, and to discuss the scheduled experiment for that day. 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 white pages will serve as the laboratory report and the duplicate 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.

The following items should be recorded in your notebook **before coming to lab** in approximately the order indicated below:

(1) Experiment number and Title
(2) Your Name, lab partner's name, lab section
(3) Date experiment is performed
(4) **Introduction** including:
    - **(5%)** Brief statement of the purpose and learning objective(s) of the experiment. What are the *new techniques* introduced by the experiment. If there is a *hypothesis* to prove, it should be included here. For example, "Does the dehydration of 3-methylcyclohexanol follow Zaitzev's Rule?"

- **(5%)** Theoretical background. Here you include a brief discussion of the theory behind the experiment. If the experiment involves a chemical reaction, a balanced chemical equation *with structures* should be shown. When applicable (particularly in CHL 202), a *reaction mechanism* should be included.

(5) **(10%)** An **organized table** listing the identity and physical properties of 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).

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

(6) **(10%)** 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.

(7) **(5%)** A **flow chart** outlining the experimental procedure.

(8) **(15%)** All **observations**, as they are made. This should consist of a complete, coherent record of every step that you perform in the experiment, followed by anything that you may have observed. This may include such things as the initiation of reflux, the observation of distillation temperature(s), the formation of two or more liquid layers, any signs of reaction such as temperature change, gas evolution, color changes, precipitation, violent reactions or anything unexpected. Any variations from the directions in the manual should also be noted. In short, write in complete sentences, everything that **you** do and everything that **you** observe.

(9) **(10%) Data and Results**, including observed melting or boiling points (distillation temperature), actual yield, theoretical yield and percent yield should be tabulated. All calculations should be shown. Note: Product yield and purity will 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.

At this point, you will have about 60% of your report completed. The following items should be added to your report before submitting it for grading:

(10) **(30%)** A **Discussion** (one-half to three-fourths page) of the experiment paying particular attention to an explanation of *why each procedure or technique was used*, e.g. “the organic layer was washed with 5% sodium bicarbonate in order to remove any acidic contaminants”, or “the organic layer was treated with saturated NaCl in order to salt-out (remove) any water”. It is not necessary to repeat items covered in the supplemental questions.

A brief discussion of your observed results and any non-human sources of error should also be included, e.g. “the observed melting point of my product was 140-146^{o}C compared to the literature value of 152-154^{o}C. This difference may be due to the presence of byproduct (speculate on what it may be) and/or incomplete drying.” "The observed 30% yield reflects a significant amount of unreacted starting material (name) and the formation of significant byproduct (name if possible). The yield may be increased by …" Try to avoid colloquial comments like “Overall, the experiment went really well.” Let your results speak for themselves. Include **references** for all literature properties (*Aldrich* catalogue, *Chemfinder*®, *Merck Index*, etc.).

(11) **(10%) 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.

All work must be done neatly, and *in ink*, with no scribbles, erasures or white-out (draw a single line through mistakes). Gross errors in spelling or grammar will be penalized on the report. Illegible or sloppily submitted reports will be returned to you for resubmission with a 10% penalty. **IMPORTANT**: Make sure that you write legibly and that you press hard when writing so the duplicate copy will be legible. The percentages in the above description are approximate.

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. 10% 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.

Grade breakdown:

(1) Lab Reports	90%
(2) Lab quizzes	10%

Any missed lab may not be made-up without a documented excuse. Only reasonable excuses for absence will be accepted. If permission is given to make up work, that work should be made-up within one week of the time the work was scheduled to be done.

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

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 necessary to perform most experiments, 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 or your breakage card(s) at the stockroom. At the end of the semester (check-out), any unused portion of your breakage card will be refunded to you. 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.

CHL 202- Laboratory for Organic Chemistry II
Spring Semester, 2016

Section 41: **Judy Straub**; Section 42: **William A. Price**
Section 43: **Laura Grande**; Section 44: **Thomas S. Straub**

La Salle University CHL 201-202 Custom Laboratory Manual from Mohrig, Hammond, Schatz and Morrill, *Modern Projects in Organic Chemistry* , 3rd edition, W.H. Freeman Custom Publishing, New York, 2010.

Date	**Experiment**
Jan. 19-21	Safety, Check-in, Introduction to Infrared Spectroscopy
Jan. 26-28	**EXP 14 (Project 5.2)** Grignard Synthesis of Secondary and Tertiary Alcohols
Feb. 2-4	**EXP 14**, continued.
Feb. 9-11	**EXP 16** (**Project 4.1):** Interconversion of 4-*tert*-Butylcyclohexanol and 4-*tert*-Butylcyclohexanone. **4.1:** Green Chemistry: Oxidation **(EXP 14 due)**
Feb. 16-18	**EXP 16 (Project 4.2)** Sodium Borohydride Reduction
Feb. 23-25	**EXP 11:** Diels-Alder Reaction **(EXP 16 due)**
March 1-3	**EXP 12:** Selectivity in the Bromination of Acetanilide **(EXP 11 due)**
March 8-10	Spring Break!
March 15-17	**EXP 13:** Acylation of Ferrocene **(EXP 12 due)**
March 22-24	**EXP 13**, continued
March 29-31	**Handout:** Microwave induced Fischer Esterification **(EXP 13 due)**
Apr. 5-7	No lab **– Wednesday, 4/6 is Special Monday**
Apr. 12-14	**EXP 17:** Synthesis of a Mosquito Repellant: *N,N*-Diethyl-*meta*-toluamide (DEET) **(Microwave lab due)**
Apr. 19-21	**EXP 18 (Project 11):** Aldol-Dehydration Chemistry Using Unknown Aldehydes and Ketones. **11.1:** Preparation and Characterization of Aldol-Dehydration Condensation Product **(EXP 17 due)**
April 26-28	**EXP 18 (Project 11): 11.2** Preparation and Characterization of Solid Derivatives of the Unknown Aldehyde and Ketone
May 3-5	Check-out. **(EXP 18 due)**