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Text: *Organic Chemistry Laboratory*, 3rd Edition, Bell, Taber and Clark,
Thomson Brooks/Cole, 2001.

Lab Notebook: *Organic Chemistry Laboratory Notebook*: this 100-page notebook is available at the Villanova bookstore.

Laboratory: T 8:30 – 11:20 am, Mendel 385/393

Contents:

This class will involve the synthesis, isolation, purification, and characterization of a variety of organic molecules. The laboratory is designed to reinforce concepts from CHM 2211 and to allow you to learn the basic techniques and skills employed by practicing organic chemists.

As a courtesy to your peers, please turn off all cell phones during lab. When your phone is needed as a timer, use a plastic baggie to cover your phone - don't use your touch screen with gloved hands!!!

General Policies:

1. *Partners:* Students will work in pairs for experiments. The instructor will assign partners at the start of each experiment. While students are encouraged to work together as laboratory partners and to discuss laboratory results as a group, all entries in your notebook must be written in your own words. It is unethical to copy pre-labs and/or other parts of your lab reports from other persons or sources.
2. *Preparedness:* Students must come to the laboratory prepared; this includes reading the laboratory handout and completing the prelaboratory assignment. Being prepared is imperative for completion of the experiments in a timely fashion and for safety reasons.
3. *On-time arrival:* At the beginning of the lab period, safety instructions and other procedures will be discussed and prelaboratory assignments will be checked for completeness. Next, students will take a quiz based on this information. Latecomers will not be permitted to submit the prelaboratory assignment, take the quiz, or conduct the experiment.
4. *Chemical use:* Common sense and consideration of fellow workers must be exercised rigorously and constantly. This includes proper disposal of chemical waste and *keeping the laboratory clean (both common areas and your work area)*. This will be an important part of your technique grade.
5. *Safety:* Students must wear mono-goggle safety glasses at all times. Experimenters must wear proper lab attire. Safety must be everyone's first priority in the laboratory. Students will be dismissed from the laboratory and assigned a zero for the lab period for *any* infraction of the safety regulations described in class and documented in the general course syllabus.
6. *Sign-out:* Once you have cleaned out your fume hood at the end of the lab, get signatures in your lab notebook before you leave for the day (yours, your partner's, and TA or instructor).

Please refer to the general course syllabus for information on attendance, grading, laboratory attire, technique grades, quizzes, laboratory notebook requirements, and a schedule of experiments for the semester.

Syllabus
CHM 2201 Organic Chemistry Laboratory I
Fall 2015

- Text (used for both Organic Chemistry Laboratory I and II): C.E. Bell, D.F. Taber and A.K. Clark, *Organic Chemistry Laboratory with Qualitative Analysis*, 3rd edition, Brooks/Cole-Thomson Learning, Pacific Grove, CA, 2001.
- A Student Laboratory Research Notebook is also required; a 100-page notebook is available at the Villanova bookstore.

Laboratory Location: Mendel 385, 393, or 283

Course Description and Objectives: Organic Chemistry Laboratory I will provide students with practical experience in using techniques associated with operational organic chemistry. The first part of the course will deal with methods, such as melting point determination, recrystallization, distillation, extraction, thin layer, column and gas chromatography and infrared spectroscopy, used to analyze, identify and purify organic compounds. These skills are crucial in isolating, purifying and analyzing the compounds that will be prepared in the latter part of the course. The reactions that will be carried out have been chosen to illustrate concepts and reactions that are covered in Organic Chemistry I lecture (CHM 2211).

Attendance:

- Attendance is required for all laboratory sections.
- There are **NO** make-up labs.
- If you miss a lab, or plan on missing a lab, for a legitimate reason (an **excused absence**, such as illness or university obligation) you need to *contact your instructor immediately to discuss your absence*.
- **Excused absences** will not be counted against you in your grade for the course.
- **Unexcused absences** will result in a grade of “zero” for all work associated with the missed laboratory session. *Multiple unexcused absences may ultimately lead to a failing grade in the course.*

Grading: You will be evaluated based on the following approximate emphasis:

- | | |
|----------------------------|------|
| 1. Lab Notebooks: | 50% |
| 2. Quizzes: | 30% |
| 3. Technique and Products: | 20%* |

* Grading may vary, depending on your instructor.

* Faculty members are responsible for maintaining the integrity of the evaluation and grading consistent with Villanova University's Undergraduate Grading System.

Academic Integrity

All students are expected to uphold Villanova's Academic Integrity Policy and Code. Any incident of academic dishonesty will be reported to the Dean of the College of Liberal Arts and Sciences for disciplinary action. For the College's statement on Academic Integrity, you should consult the [Enchiridion](#). You may view the university's Academic Integrity Policy and Code, as well as other useful information related to writing papers, at the Academic Integrity Gateway web site: <http://library.villanova.edu/help/academicintegrity>

Laboratory Attire:

1. **Approved safety goggles must be worn at all times while in the lab during the lab period.** They must be worn over the eyes, not on the forehead.
2. **No tank tops.** Aprons provide little protection above the waist. Students must wear shirts that cover the majority of the torso. Oversized or baggy shirts and sweaters are not appropriate.
3. **No exposed skin below the waist.** *Unacceptable clothing includes* (but is not limited to): shorts, skirts, Capri pants, tights, leggings, 'jeggings', yoga pants, 'skinny' jeans, pajama pants, sandals, open-toe shoes (or any shoes in which the top of the foot is exposed – e.g., ballet flats), etc.

Appropriate attire for a chemistry laboratory course might include: t-shirt, jeans, and sneakers / tennis shoes *with socks*.

Regarding points 2 and 3, students in violation of **either** of these rules **are not allowed in the lab; no exceptions will be made.** Students may be given the option of returning to lab after they have changed into proper attire, but they will not be given extra time at the end of the period to make up the lost time or to complete assignments.

Students who are in violation of attire / behavior rules or those outlined in the safety contract that is signed at the beginning of the semester may be issued a warning (student signature required). Repeat violations will result in dismissal from the lab for the period with an unexcused absence (grade of 0) recorded for the period. The Laboratory Absence (Attendance) Policy will apply in these cases.

Technique Grades

Technique grades are based on:

- Preparation for the lab
- Observance of **SAFETY** guidelines – safety glasses worn at all times after lesson introduction is finished; wear proper attire; safe handling of chemicals. See the Laboratory Attire policy (above). Always replace caps on reagent bottles after finished using the reagents; don't leave the cap off for the next person. Comply with safety rules as outlined in front of your laboratory notebook.
- Cleanliness of your bench/hood areas as well as common areas used by the entire lab (weighing areas, hood, etc.). If your hood/bench area is not clean after you leave the lab for the day, your technique grade for the day will be adversely affected! Likewise, if the community hood and balance areas are not clean at the end of the lab, the entire class's technique grade will be lowered. If you spill something in the hood or at the balance area, immediately clean it up.
- Effort, proficiency and neatness in setting up equipment, carrying out experiments, collecting data and keeping up to date lab records. Do not record data on scraps of paper and then add to your notebook. All data is to be recorded directly in notebook.
- Teamwork: Students may be working in teams of two to four for some of the experiments carried out during the semester. Each team member will be expected to make a significant contribution to successfully performing the experiment. Successful and smoothly operating teams are able to complete experiments in an efficient manner by having each team member assigned to specific tasks in preparation for performing the main experiment(s).

Technique Grades (continued)

- Mastery of new lab techniques: Read the text carefully and pay attention during the lab introductory lecture so that you have some familiarity with new lab techniques before you perform the experiment.

Quizzes

There will be quizzes during the semester. Depending on your instructor, quizzes may cover material from the lab text, the introductory laboratory lecture and/or from the experiment carried out in the lab. The quizzes may be open or closed book, again depending on your instructor.

Laboratory Notebook Requirements

All write-ups must be placed in a Student Laboratory Research Notebook, available along with your text, in the Villanova Bookstore. Use a ballpoint pen for writing and place all information directly in the notebook as it is obtained. Leave two pages at the beginning for a Table of Contents. It is important that you write clearly and legibly. However, since some writing must be done in the laboratory, you will not be expected to provide polished, formal reports. Note that only the tear-out copies from the notebook are to be handed in for marking. The original copies are to remain in your possession.

Each experimental write-up will consist of three parts:

1. A **Pre-Lab Write-up**, which you must complete before lab and hand in to your TA when entering the lab.
2. Your **Observations** recorded in lab.
3. Your **Conclusions**, which you complete during or after lab (as directed).

A typical **Example of a Laboratory Write-up** will be provided as a handout along with a grading rubric titled, **Grading for CHM 2201 Laboratory Notebooks**. Note that your Instructor or TA may also have additional items that they want to be included in experimental write-ups.

► **The Pre-Lab Write-up:**

On entering the lab, you must present the **notebook copy** of your pre-lab write-up for that day to your TA. The purpose of the write-up is to demonstrate reasonable preparation for, and knowledge of the experiment to be conducted. If you do not demonstrate such knowledge, either written and/or orally, you will not be allowed to carry out the experiment. It is imperative that you complete the reading assignments and compose your own pre-lab reports. **It is unethical to copy pre-labs, and other parts of your lab reports from other persons or sources. It is also a violation of the Code of Academic Integrity of the College of Liberal Arts and Sciences.**

The Pre-Lab Write-up should include:

1. Your **Name, Course, Section Number, Lab Partner** (if any) and **Date** (at the top of each page).
2. **Title of the Experiment**
3. **Statement of Purpose** of the experiment (a sentence or so regarding the chemical syntheses, techniques and principles that the experiment demonstrates).
4. **Reaction Sequence** (where applicable). Reactions should be written in standard manner with solvent and temperature information recorded below reaction arrows, and the stoichiometry clearly indicated. Individual steps of multistep processes are to be included.
5. **Table of Physical Constants** of reagents to be used in the experiment. Information (such as boiling points and densities for liquids, melting points for solids, molecular weights, solubilities, etc.) for completing the table may be found in the lab text (Appendix C, page 283), in the CRC handbook, in an Aldrich Chemical Catalog, copies of which are in the laboratory, or at the Aldrich Chemical web site with the following internet address: <http://www.sigmaaldrich.com/chemistry/chemical-synthesis/chemical-synthesis-catalog.html>
6. **Safety.** Include a description of safety concerns for all chemicals to be handled, describe safety precautions that should be taken, and make note of any technique-specific safety concerns. To find this information, you may use the same resources described above to prepare the Table of Physical Constants. If you wish to learn more about the reagents we are using in the lab, you may utilize the ChemWatch program, which is a database of safety datasheets (SDS). The link to ChemWatch may be found on this page within the Department of Chemistry website: <https://www1.villanova.edu/villanova/artsci/chemistry/Resources/software.html>
7. **Procedure.** The procedure indicated in the lab text and/or from a handout should be recorded in **brief outline format**. This step-by-step outline should include all information required for conducting the experiment, such as procedures, amounts of chemicals or solvents to use, reaction times and temperatures, and other important considerations (such as “until the color fades”, or “add 11.0 M sodium hydroxide with stirring, to pH 9.0”). Refer to the handout **Grading for CHM 2201 Laboratory Notebooks** for further guidance.

► **Observations** (to be recorded in lab):

Observations should be recorded directly in your notebook as you carry out your experiment. Keep a running record of your observations and make sure it is clear how the observations correlate to the steps in the procedure. Make a note of any modifications to the procedure that differ from what you outlined in your notebook before coming to the lab. Data (measured masses, volumes, etc.) should be recorded directly in your notebook as you acquire the information. Your instructor may prefer a numbered list, a bulleted list, a two-column format (one column for the procedure, and one column for in-lab observations), or another format – find out the formatting your instructor expects for this section.

► **Observations** (to be recorded in lab), (*continued*):

Redraw the reaction sequence, only this time insert beneath the structures the quantities of reagents actually used in the experiment. Refer to the handout **Grading for CHM 2201 Laboratory Notebooks** for many examples of typical information that you will record in your notebook. Attach copies of spectra (IR, NMR, etc) and GC traces to your notebook pages for inclusion in your submitted lab report. IN OTHER WORDS, RECORD EVERYTHING THAT YOU DO IN ENOUGH DETAIL SO THAT ANOTHER PERSON WITH A BASIC KNOWLEDGE OF CHEMISTRY COULD REPEAT YOUR EXPERIMENTAL WORK.

Do not use personal pronouns (I, we, he, she, us, etc.) when writing in your notebook. Instead of writing “I added 4 grams of phenol to 30 mL of methanol in a 100 mL round bottom flask equipped with a reflux condenser” write “To a 100 mL round bottom flask containing 30 mL of methanol and fitted with a reflux condenser was added 4 g of phenol.”

Notebook pages containing your observations must be initialed and dated by your professor or TA before you leave lab for the day.

► **Conclusions** include:

1. Results, such as % yields (show calculations) and comparisons between literature values and measured values for yields, melting points and boiling points, chromatographic, and spectral data. Interpretation of infrared and NMR spectra.
2. Your interpretation of unusual results and/or results not in agreement with those indicated in the lab text or by the instructor. You should focus especially on how errors or other factors might have contributed to the outcome.
3. Your evaluation of the experiment, and a brief summary of how you might improve your procedure if you were to perform it again.
4. Answers to assigned post-lab questions (if any), and answers to any **yellow highlighted questions** from the detailed experimental procedure (include question numbers for clarity).

Observations and Conclusions must be handed in for grading at your next lab session. Points will be deducted for late submissions.

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**Office of Disabilities and Learning Support Services:**

Students with disabilities who require reasonable academic accommodations should schedule an appointment to discuss specifics with me. It is the policy of Villanova to make reasonable academic accommodations for qualified individuals with disabilities. You must present verification and register with the Learning Support Office by contacting 610-519-5176 or at [learning.support.services@villanova.edu](mailto:learning.support.services@villanova.edu) or for physical access or temporary disabling conditions, please contact the Office of Disability Services at 610-519-4095 or email [stephen.mcwilliams@villanova.edu](mailto:stephen.mcwilliams@villanova.edu). Registration is needed in order to receive accommodations.

## Schedule of Laboratory Experiments

| Lab/<br>Week # | Month        | Day/Date |    |    |    |    |    | Experiment/Activity                                                                                                                                                 | Lab Text <sup>1</sup><br>Pages                   |
|----------------|--------------|----------|----|----|----|----|----|---------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------------|
|                |              | M        | Tu | W  | Th | F  | M  |                                                                                                                                                                     | Carey &<br>Giuliano <sup>2</sup><br>Pages        |
| 1              | Aug          | 24       | 25 | 26 | 27 | 28 | -  | Check-in, Safety, Melting point determination,<br><b>Exps. 2A, C</b>                                                                                                | 17-22                                            |
| 2              | Aug/<br>Sept | 31       | 1  | 2  | 3  | 4  | -  | Preparation/Recrystallization of Acetanilide,<br><b>Exp. 3C</b>                                                                                                     | 23-32                                            |
| 3              | Sept         | -        | 8  | 9  | 10 | 11 | 14 | Gas Chromatography (GC), <b>Exp. 6A</b> ;<br>Thin Layer Chromatography (TLC), <b>Exp. 8B</b>                                                                        | 59-65<br>73-79                                   |
| 4              | Sept         | -        | 15 | 16 | 17 | 18 | 21 | Simple Distillation, Fractional Distillation,<br>Fraction Analysis using Gas Chromatography<br>(GC), <b>Exps. 5B, 5C</b>                                            | 47-58<br>59-65                                   |
| 5              | Sept         | -        | 22 | 23 | 24 | 25 | 28 | Molecular Models, Conformation and<br>Stereochemistry<br>(Handout provided)                                                                                         | 96-119<br>180-183<br>262-288                     |
| 6              | Sept/<br>Oct | -        | 29 | 30 | 1  | 2  | 5  | Isolation of Caffeine from Tea,<br>Week 1 – Extraction and TLC <b>Exp. 7B</b>                                                                                       | 67-72<br>(33-46)                                 |
| 7              | Oct          | -        | 6  | 7  | 8  | 9  | 19 | Isolation of Caffeine from Tea,<br>Week 2 – Column Chromatography <b>Exp. 7B</b>                                                                                    | 67-72<br>(33-46)                                 |
| *****          | Oct          | 12       | 13 | 14 | 15 | 16 |    | Semester Recess – no lab                                                                                                                                            |                                                  |
| 8              | Oct          | -        | 20 | 21 | 22 | 23 | 26 | Infrared (IR) Spectroscopy; Demo of how to<br>use IR Spectrophotometer for Solids &<br>Liquids; Determination of Molecular Structure<br>of Unknown, <b>Exp. 11E</b> | 93-106<br>545-552                                |
| 9              | Oct/<br>Nov  | -        | 27 | 28 | 29 | 30 | 2  | Dehydration of Alcohols, <b>Exp. 20B</b>                                                                                                                            | 205-209<br>189-197                               |
| 10             | Nov          | -        | 3  | 4  | 5  | 6  | 9  | Properties of Hydrocarbons, <b>Exps. 16A, B, D,</b><br><b>E, F</b>                                                                                                  | 183-185<br>Ch 2,<br>Ch 6.6, 6.10,<br>11.12, 12.4 |
| 11             | Nov          | -        | 10 | 11 | 12 | 13 | 16 | Nucleophilic Substitution of Alkyl Halides,<br><b>Exps. 17A, B, C</b>                                                                                               | 187-192<br>306-329                               |
| 12             | Nov          | -        | 17 | 18 | 19 | 20 | 23 | Diels-Alder Reaction, <b>Exp. 32A</b>                                                                                                                               | 269-273<br>391-396                               |
| *****          | Nov          | -        | 24 | 25 | 26 | 27 | -  | Thanksgiving Recess – no lab                                                                                                                                        |                                                  |
| 13             | Nov/<br>Dec  | 30       | 1  | 2  | 3  | 4  | -  | Checkout                                                                                                                                                            |                                                  |

1. [CHM 2201/2202 Organic Chemistry Laboratory Text](#): C.E. Bell, D.F. Taber and A.K. Clark, *Organic Chemistry Laboratory with Qualitative Analysis*, 3<sup>rd</sup> edition, Brooks/Cole-Thomson Learning, Pacific Grove, CA, 2001.
2. [CHM 2211/2212 Organic Chemistry Lecture Text](#): F.A. Carey and R.M. Giuliano, *Organic Chemistry*, 9<sup>th</sup> edition, McGraw-Hill, New York, NY, 10020.