ORGANIC CHEMISTRY LABORATORY Chemistry 314 summer 2023

Coordinator and Lecturer: Dr. T. Fadeeva

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REQUIRED COURSE MATERIALS

- 1. Text: Macroscale & Microscale Organic Experiments by Williamson (6th ed). Custom Version for Rutgers University.
- **2. Laboratory Notebook:** A hardcover or soft cover notebook with numbered gridded pages and tear out carbon-copy duplicate pages is required (spiral/loose-leaf notebooks are not acceptable) for recording laboratory work. They are available in the Rutgers University Bookstore.

3. Safety goggles

To protect your eyes in the laboratory (must be face-fitting and form seal around eyes; ordinary glasses or other types of safety glasses including contact lenses worn under goggles are not acceptable). Goggles are available at bookstores.

COURSE POLICIES

- 1. The theoretical part of each experiment will be discussed in a prelab lecture.
- 2. Read the material and be prepared before lecture and lab.
- 3. A grade for each experiment is based on your report. So, it's crucial to submit a report on time (see the schedule). **3 points will be subtracted for each day after the deadline.**
- 4. MAKE-UP: A missed lab period may be made up only for valid medical reasons and with a note from the dean's office. Dr. Fadeeva's approval is required for a makeup. Make sure to contact me immediately when you miss a lab.
- 5. Lectures: Attendance in lectures is essential in order for a student to do well in this course. Significant amounts of additional material, including modifications to the lab procedure is provided in the lecture. Much of the quiz and exam material as well as what is required in the lab report will be provided during the lecture for the course in conjunction with the textbook. Lab instructors will assume that students have attended the lecture and will not provide information that has already been provided in the lecture.
- 6. **Quizzes:** a short quiz (10 mins) will be given in the lab at the beginning of the lab period on the dates indicated. A safety quiz will be given on the first day of the lab.
- 7. **A Final Exam** will be given on the specified date. The material for the exams will come from your lab experiments, readings and lecture material.
- 8. **Laboratory Safety:** All people in the lab must observe the safety rules. We'll discuss safety precautions for each particular experiment.

- 9. Students with Disabilities: If you have a disability, you are urged to speak to the course supervisor to make the necessary arrangements to support a successful learning experience. Also, you must arrange for the course supervisor to receive a letter from your College's Disability Concerns Coordinator verifying that you have a disability. The student must contact the Office of Disability Services to determine his/her Coordinator (848-445-4477 or <u>https://ods.rutgers.edu</u>.
- 10. Academic honesty: You are being graded on the work you perform. Use of lab reports from other students (past or present) is expressly forbidden. Both the lender and the borrower are subject to severe penalties. Some discussion about the labs is acceptable at the discretion of the lab instructor, but you must perform all the work (including the data analysis and answering of questions) yourself. A lab report is NOT a collaborative effort- it must be written in your own words, using your own data. The lab instructor is free to ask you at any point to explain what you are doing. This is to help the lab instructor instruct the confused and prevent copying of answers. If you are confused, ask for help. Don't just copy an answer. Academic honesty also applies to all quizzes and exams in this course.

Unauthorized posting and sharing of course material (including but not limited to: syllabi, lecture notes, past, present and future quizzes and exams, prelab and postlab questions) in paper form or online during the current semester and/or in the future is STRICTLY PROHIBITED. The unauthorized posting and sharing of course material is a violation of copyright law AND a violation of academic integrity and appropriate action will be taken against person(s) who violate this law. Report any violations promptly.

11. **Chain of Command:** If you have a question about grading, you should first talk about it with your lab instructor <u>within the following lab period after receiving your grade.</u> If you are not satisfied with the explanation, you may raise the question with the course coordinator. We will not intervene for questions of a small number of points. Decisions made for safety (such as ejection for violation of safety rules) can be made by any lab instructor, the stockroom personnel, or the coordinator, and these decisions are final and not subject to appeal. If you have a question about content, concepts or procedures then you may ask any lab instructors or the coordinator for help.

List of Experiments:

- 1. Nucleophilic Substitution.
- 2. Esterification. Synthesis of n-butyl acetate.
- 3. The Borohydride reduction of a Ketone. Hydrobenzoin from benzil.
- 4. Aldol condensation. Synthesis of dibenzalacetone.
- 5. Benzoin condensation. Catalysis by the cyanide ion and Thiamine.

GRADING

1.	Laboratory Reports (60 points x 5 experiments)	300 points
2.	Safety Quiz	5 points
3.	Quizzes (12 points x 5)	60 points
4.	Lab Instructor evaluation	25 points
5.	Written Exam	110 points
	TOTAL	500points

GUIDELINES FOR WRITING LAB REPORTS IN LABORATORY NOTEBOOK

The laboratory notebook is the primary record of the work you accomplish in the laboratory. It should be well organized, legible, and an accurate record of your work. You can have a report handwritten (legible!) or typed, **BUT a procedure/observation, calculations and a sketch MUST BE handwritten**.

- 1. Use a hardcover or soft cover bound composition book with numbered pages and tear-out carbon-copy duplicate pages. Write your name, address, course number, section number and lab instructor's name on the cover.
- 2. Start a new experiment on a fresh page. Make all entries in ink never in pencil.
- 3. Hand-write directly in the notebook not on scraps of paper; do not tear out pages (except to hand in laboratory reports to lab instructor there MUST always be the duplicate record remaining in your lab notebook). Record a complete, accurate account of all experiments and results. Show all calculations. Follow guidelines for writing a laboratory report shown below.
- 4. Never erase incorrect entries; merely draw a line or two through blank spaces and/or incorrect words so that they are still legible.

Guidelines for writing a laboratory report

The laboratory report is divided into 2 parts:

- I. Pre-laboratory notes + procedure. (must be completed before the lab.)
- II. In-laboratory data + Post-lab (to be turned in at the beginning of the next laboratory period EXCEPT when noted)

The entire report should be no longer than 4-6 handwritten pages using the format shown below.

I. PRE-LABORATORY NOTES & IN-LABORATORY DATA

A. Pre-laboratory notes -

Guidelines for writing the Prelab Notes for Each Experiment (NOTE: YOU MUST HAVE THE PRELAB AND PROCEDURE WRITTEN UP BEFORE YOU COME INTO THE LAB FOR EACH OF YOUR LABORATORY SESSIONS). YOUR LABORATORY INSTRUCTOR WILL NOT ALLOW YOU INTO THE LABORATORY UNLESS YOU HAVE THE PRELAB NOTES AND PROCEDURE COMPLETED The pre-laboratory notes should include:

1) Title of Experiment: You will have to write the title of the experiment and follow it with a reference.

References:

If you used the procedure from the textbook, then the Reference would be the pages of the book from which you wrote the procedures, followed by the author's name and the title of your textbook.

Example: Recrystallization (Reference: pg. 64-65) Williamson, K. Macroscale and Microscale Organic Experiments

If you used a handout, then the Reference would be "Chemistry 314 Handout".

2. Prelab questions (if any)

3. A <u>very brief</u> statement about the purpose of the experiment - (1-2 lines)

4. An Introduction section that discusses briefly the technique being used (purpose and use) and/or the reaction being carried out (in a synthesis reaction). The information needed for this section is typically found in the text and the lecture.

5. Pertinent balanced equation(s) for the reaction(s) using structures and names of reactants and products. Equations for important side-reactions should also be included.

6. A sketch of the apparatus set-up when a new technique is being used.

7. Chemical Hazards of the chemicals you are using and Precautions to be taken with the chemicals being used: Information can be obtained using the MSDS on canvas.

8. Table of reagents and/or reactants and/or products - this table will vary somewhat with the nature of the experiment. It must include literature values (if you cannot find these in your

textbook, you can find it in The CRC Handbook of Chemistry and Physics or The Merck Index or other online sources).

When the experiment involves a synthesis, you must have <u>Table for the Reactants</u> (or you can incorporate the reactants into the table of reagents) which should include:

(a) Physical properties (as mentioned above)

- (b) Molecular weight
- (c) Amount used in grams or mL
- (d) Moles used

When appropriate, you should identify the Limiting Reagent.

When conducting a synthesis, you should also have a <u>Table for the Product(s)</u>, which should include:

(a) Physical properties (mol. wt, mp, bp, density, solubility) of product and side product(s)

(b) Molecular weight of each product

(c) Theoretical weight in grams of only expected product (show calculations)

8. <u>Brief outline</u> of expected experimental procedure. <u>PUT THIS PART ON A</u> <u>SEPARATE PAGE.</u> You should divide the page into two columns. Write the outline on the left side of the page. The right side of the same page can be used for recording the In-laboratory observations and comments that are pertinent to each procedural step. Make sure that the procedure is brief!!

In Laboratory Data

In-laboratory data should include:

1. The code letters or numbers of any assigned unknowns.

2. A precise record of all experimental data including weights, volumes, boiling point ranges, melting point ranges and other measurements pertinent to the experiment.

Your observations and comments for each procedural step. These should include changes in color, texture, clarity of solution, temperature, smell etc.

NOTE: A large number of points for each lab report will be attributed to the observations you make. Experimental work depends VERY heavily on accurate observations.

An example of observations is shown in the procedure table below.

Procedure	Observations
1. Place 42 mg benzil, 42 mg	Benzil: yellow crystalline solid,
1,3 diphenylacetone and 0.4	Weight: 40.5 mg
mL triethylene glycol in a	1,3-diphenylacetone: white powdery solid,
test tube.	Weight: 41 mg
2. Clamp tube over hot sand	Triethylene glycol: clear, colorless, viscous
bath, insert thermometer and	liquid
heat until benzil is dissolved.	Volume: 0.4 mL
	Appearance of mixed solution: benzil forms a yellow suspension in clear solution
	Benzil dissolves in solution when heated to
	form yellow clear solution at 120°C

Note: The Prelab notes must be turned in to your lab instructor at the beginning of the lab period and the In-Lab data must be turned in to your lab instructor and the end of the laboratory period. Failure to turn in the prelab at the appropriate time may result in significant deduction of points or it not being graded. II. POST LAB

Post Laboratory Reports should include the following:

1. Post Lab Questions. The list of post lab questions is listed in the syllabus in the Post lab Assignment section.

2. <u>Analysis of experimental data</u> - this will vary with the type of experiment. Include when appropriate:

(a) Specified calculations using experimental data. For experiments involving a synthesis, the percent yield of the product(s) should be calculated.

(b) Graphs using data from experiments and an analysis of graphs

(c) Results from analytical techniques like TLC, melting points, IR, NMR and explanations of results. (This can also be shown in the discussion section).

3. A discussion of these results.

(a) Each observation in the in-lab report should be explained. If an observation was a result of a chemical reaction, the balanced equation and mechanism (if applicable) should be used to explain the observation.

Example: When phenyl magnesium bromide was added to dry ice, there was initial fizzing until the excess dry ice evaporated. The reaction is represented by the following equation and mechanism.

 $PhMgBr \ + \ CO_2 \ \rightarrow \ PhCO_2^{-} \ ^+MgBr$

(b) In experiments involving synthesis, techniques used must be explained (Example: Simple distillation was used to separate cyclohexanone from cyclohexanol and water. Simple distillation was used because cyclohexanone and cyclohexanol have a large boiling point difference).

(c Comparison of products to accepted literature values - conclusion derived from the deviation from or similarity to literature value.

(d) Possible sources of error to explain why product yield deviated from theoretical yield

4. A statement regarding the conclusions that can be drawn from these results.

Make sure that you turn in the postlab questions to your lab instructor at the beginning of the subsequent lab period.

3 points will be subtracted for each day after the deadline.

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Schedule

Data	<u>Lecture</u>	Experiment	Quiz	Report is due
7/11	Nucleophilic Substitution	Safety orientation	Safety	
7/12		Nucleophilic Substitution		
7/13	Esterification, IR		Nucleophilic Substitution	
7/18		Esterification		Nucleophilic Substitution
7/19	Reduction		Esterification	
7/20		Reduction		Esterification
7/25	Aldol Condensation		Reduction	
7/26		Aldol Condensation		Reduction
7/27	Benzoin Condensation		Aldol Condensation	
8/1		Benzoin Condensation		Aldol Condensation
8/2			Benzoin Condensation	
8/3				Benzoin Condensation
8/8	Final exam			
8/9	Grading			
8/10	Grading			
8/15	Grades posted			
8/16				