Organic Chemistry Lab II
CHE-243

Lab Manual

Prepared by Ralph Fleming, 2017

Table of Content

Notice

Citation

Fleming, R. (2017). *Organic Chemistry Labl I, CHE-243, Lab Manual, 1st Ed. Medford, NJ.*

Other source

www.chemistry-solutions.com

Rowan College at Burlington County
Spring 2017

STEM Division
Syllabus: Organic Chemistry Lab II, CHE243

One Credit

Co-requisites: CHE241 Class Meeting Days: Three hours, Once per week per section,
Location: LAB SB212

Instructor(s) Ralph Fleming
Contact Information: rfleming@rcbc.edu cell: 609-354-2179
Office Hours: Mondays 10:00AM to Noon

SECTION 1:

Course Description:
This is the second semester of a two semester transfer organic chemistry lab course, for one credit each. The primary objective of the course is the hands on synthesis, purification, and characterization of organic compounds. Students prepare a research quality notebook of their laboratory work.

Laboratories:
Three hours in the laboratory per week. Laboratory preparation information, laboratory safety information, laboratory procedures, and data are entered for each experiment in a laboratory notebook per procedures outlined in this syllabus. An analysis and an abstract are written for each experiment.

Required Text and other Materials

Laboratory Text: On Blackboard for course

Required Material: Lab coat, ¾ length (available in College Bookstore).

Course Learning Outcomes

Upon completion of the course, students will be able to:

- Demonstrate safe and effective work habits in the laboratory.
- Synthesize and purify organic compounds.
- Operate laboratory equipment with proper technique.
- Construct laboratory reports of their experiments.

General Education Outcomes.

Written and Oral Communication: Communication
* Students will conduct investigative research which demonstrates academic integrity, originality, depth of thought, and mastery of an approved style of source documentation.

Scientific Knowledge and Reasoning: Science
* Students will demonstrate critical thinking skills the analysis of scientific data

Core Course Content

1. A typical sequence in oxidation reactions
2. Friedel-Crafts synthesis
3. EAS reactions
4. Esterification reactions
5. Grignard reactions
6. Intellectual property in the pharmaceutical industry

See RCBC Hand Book http://www.rcbc.edu/publications

SECTION 2: Course Structure

Goals: to introduce the student to:

1. the techniques used in synthesis of organic molecules
1. multi-step synthesis in the preparation of organic molecules
1. safe laboratory practices
1. the structure and writing of a research quality laboratory notebook.

Rationale:

Further study in the fields of biology, biotechnology, medicine, and chemistry requires the student to use his/her knowledge of the basic structure of organic molecules and organic reaction mechanisms to analyze situations and experiences that he/she encounters in the other courses and work experiences in these fields.

Course and Classroom Policies

Preparation

It is important that you come prepared for every experiment. Prepared means that you have completed the required sections of your journal such as the Physical Constants Sheet and CHP before you enter the Lab. Your instructor prior to you starting you lab work will check the lab journal. Deductions will be made for incomplete work. If you are not prepared, you will not be able to start the lab until you are prepared and your journal is complete. If you arrive late and /or not prepared there is a grade penalty and possibility forfeiture of lab period.

Attendance/ Grade Policy

1. chronically arriving late to lab 10% grade penalty per instance

2. missing lab, no makeup, zero (0) grade for that part of the experiment.
3. missing lab, makeup in another section, 10% grade penalty for the experiment.
4. missing lab, no makeup, use instructor data for report, 15% grade penalty for experiment.
5. late notebook, 1 week, 20% grade penalty for the experiment

Behavior Policy

1. Smart phone are permitted as calculators and timers.
2. Texting and phone conversations are not permitted during the lab period.
3. Students can leave the lab for emergency communications.

	Points (%)
Experiment 9	22.50
Experiment 10	22.50
Experiment 11	22.50
Experiment 12	22.50
Intellectual Property	10.00
TOTAL	100.00

Criteria for Grade Determination

Assessment Methods

Academic Honesty:

Students are expected to comply with all college policies regarding academic honesty. Acts of cheating, plagiarism or other acts of academic dishonesty are grounds for failure of the course, suspension from the college, or both. The lab journal represents your work in the course. To copy someone else's lab book information is academic dishonesty. Do not share your lab books or information. Any information obtained from another source other than yourself must be documented as to the source. See RCBC Hand Book http://www.rcbc.edu/publications

Grading: Total grade is based on the content and evaluation of the laboratory notebook per:

Grading Rubric

Grading Rubric

PRELAB exp 9		GRADE	POSTLAB		GRADE
ATTENDANCE			ATTENDANCE		
GP	2		EXPER PRO	5	
CHP	2		EXPER CALCS	7	
Physical Constants	4		Flow sheet	8	
PRELIMINARY CALCS	4		DATA TABLE	10	
RX Equation	1		ANALYSIS	15	
Data Table	1		Abstract	15	
Tech: TM, Saf, Prep	7		Tech:TM,Prep	10	
Flow sheet	2		IR	5	
Exp procedure	2				
total	25		*total*	75	

PRELAB exp 12		GRADE	POSTLAB		GRADE
ATTENDANCE			ATTENDANCE		
GP	2		EXPER PRO	10	
CHP	2		EXPER CALCS	8	
Physical Constants	4		Flow sheet	8	
PRELIMINARY CALCS	4		DATA TABLE	10	
RX Equation	3		ANALYSIS	14	
Data Table	1		Abstract	15	
Tech	5		Tech	5	
			IR	5	
Exp procedure	4				
total	25		*total*	75	

PRELAB exp 10		GRADE	POSTLAB		GRADE
ATTENDANCE			ATTENDANCE		
GP	2		EXPER PRO	10	
CHP	2		EXPER CALCS	8	
Physical Constants	4		Flow sheet	8	
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RX Equation	3		ANALYSIS	14	
Data Table	1		Abstract	15	
TECH	5		Tech	5	
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PRELAB exp 11		GRADE	POSTLAB		GRADE
ATTENDANCE			ATTENDANCE		
GP	2		EXPER PRO	10	
CHP	2		EXPER CALCS	8	
Physical Constants	4		Flow sheet	8	
PRELIMINARY CALCS	4		DATA TABLE	10	
RX Equation	3		ANALYSIS	14	
Data Table	1		Abstract	15	
prep/tech	5		Tech	5	
			IR	5	
Exp procedure	4				
total	25		*total*	75	

Intellectual Property: Report:10 %, Presentation: 90%

For further information on grading, See: http://www.chemistry-solutions.com/

Course Schedule or Calendar

See: http://www.chemistry-solutions.com/

Week 1 : Introductions, Safety, Review
Weeks 2-4: Experiment 9

Weeks 5-7: Experiment 10
Weeks 8-10: Experiment 11
Weeks 11-13: Experiment 12
Week 14: Intellectual Property

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Weeks 5-7: Experiment 10
Weeks 8-10: Experiment 11
Weeks 11-13: Experiment 12
Week 14: Intellectual Property

SECTION 3: College Resources

College Policies

In order for students to know their rights and responsibilities, all students are expected to review and adhere to all regulations and policies as listed in the College Catalog and Handbook. These documents can be accessed at rcbc.edu/publications . Important policies and regulations include, but are not limited, to the following:

- College Attendance Policy
- Grading Standards
  - Withdraw (W) and Incomplete Grades (I & X)
  - Withdrawal date for this semester **(will vary)**
- Student Code of Conduct
  - Academic Dishonesty/Plagiarism and Civility
- Use of Communication and Information Technology

Office of Student Support and Disability Services

RCBC welcomes students with disabilities into the college's educational programs. Access to accommodations and support services for students with learning and other disabilities is facilitated by staff in the Office of Student Support (OSS). To receive accommodations, a student must contact the OSS, self-identify as having a disability, provide appropriate documentation, and participate in an intake appointment. If the documentation supports the

request for reasonable accommodations, the OSS will provide the student with an Accommodation Plan to give to instructors. For additional information, please contact the Office of Student Support at 609-894-9311, ext. 1208, disabilityservices@bcc.edu, or rcbc.edu/studentsupport .

Educational Technology Statement

Rowan College at Burlington County (RCBC) advocates the use of technology to enhance instruction. Students should assume that classroom and online technology will be used throughout their coursework at RCBC, as it will most certainly be used in their future education and careers. The College provides on-campus facilities for the convenience of the RCBC community. Various college departments, including the Office of Information Technology and the Office of Distance Education, provide technology training and assistance to faculty and students.

Student Success Services

RCBC offers a variety of free services for its students including those listed below. Descriptions of these services, as well as many others, can be found in the College Catalog and Handbook and on the RCBC website at rcbc.edu/publications .

- Academic Advisement (rcbc.edu/advising)
- Career Services (rcbc.edu/careers)
- Educational Opportunity Fund (EOF) (rcbc.edu/eof)
- Financial Aid (rcbc.edu/financialaid)
- International Students Office (rcbc.edu/international)
- Library/Integrated Learning Resource Center (ILRC) (rcbc.edu/library)
- Office of Veteran Services (rcbc.edu/vets)
- Student Support Counseling (rcbc.edu/cpit)
- Tutoring Center (rcbc.edu/tutoring)
- Test Center (rcbc.edu/testcenter)
- Transfer Services (rcbc.edu/transfer)

Grading

Course and Classroom Policies

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Tech	5		Tech	5	
			IR	5	
Exp procedure	4				
total	25		***total***	75	

ORGANIC CHEMISTRY LABORATORY

KEY ELEMENTS TO CONSIDER IN REPORTS:

ABSTRACT:
Does it have the proper scope and depth for an abstract?
Would someone reading this abstract know what was done? Why it was done? What is the conclusion? In other words, does the abstract include the objective of the research, results, and a concluding statement?
Does it include data and facts and avoid subjective terms?

ANALYSIS:
It is comprehensive?
Does it propose reasonable explanations for yield and purity results in synthesis experiments?
When applicable, does it address the mechanism of the reaction?
When applicable, does it address side products? Purification techniques?

EXPERIMENTAL PROCEDURE:
Does it include enough detail that someone to reproduce their work and obtain the same results? Does it contain key observations, dates, and conditions? , Does it contain any modifications to, or elements not explicit ion the General Procedure?

SPECTRA/CHROMATOGRAMS:
Are all required spectra/chromatograms present?
Are they clearly labeled?
Do they contain required sample preparation and instrument data?
Are key elements identified and compared to standards?

DATA TABLES:
Are they comprehensive and neat?
Do they contain the proper units when required?
Does the data table serve to help the reader understand the results and conclusions?

FLOW SHEETS:
Does it accurately diagram what was done in the experimental procedure?
Are all reagents, side products, and final products accounted for?

CALCUALTIONS:
Are all required calculations present? There should be one example of each unique calculation.
Are they accurate, clearly labeled, and include the proper units?

TECHNIQUE (LAB):
Does the student work safely, wear PPE, keep lab clean, and dispose of waste properly?
Does the student effectively use instrumentation and lab equipment?

TECHNIQUE (LAB BOOK)
Is the report neat? Does it follow the protocol for keeping research quality notebooks?

GRADING OF LABORATORY LAB NOTEBOOK: ANAYSIS, ABSTRACT, TECHNIQUE
CHE-241, CHE-243

The **Analysis** section of the notebook is evaluated based on the following:

1. Form: format, header, title, paragraphs are used to separate points.
2. Function: the writing is readable, organized, logical order, free of mistakes, proper grammar, and punctuation. Avoid informal wording, addressing the reader directly, and jargon, or slang. Use concrete terms. Do not use pronouns.
3. Content: all parts of the experiment should be discussed and analyzed. Key elements reported, (percent yield, purity, IR, MP, RI, relative comparison to Literature or standard values, note compounds, structures, impurities, and any relationships). Do not include procedures.
4. Conclusion/summary: A strong affirmative conclusion or summary statement. Identify structures or compounds.

The **Abstract** section of the notebook is evaluated based on the following:

1. Form: Abstract format: citation, header, author, and title. Written in the past tense. A single paragraph, no more than three. Be concise.
2. Function: the writing is readable, organized, logical order, free of mistakes, proper grammar, and punctuation. Avoid informal wording, addressing the reader directly, and jargon, or slang. Use concrete terms. Do not use pronouns.
3. Content: Key elements reported, (percent yield, purity, IR, MP, RI, relative comparison to Literature or standard values, note compounds, structures, impurities, and any relationships).
4. Conclusion/summary: A strong affirmative conclusion or summary statement. Identify structures or compounds.

An abstract is a concise summary of the completed experiment. In a minute or less a reader can learn the rationale behind the experiment, the general procedure, pertinent results, and the conclusions of the experiment.

The Technique evaluation: Laboratory notebook preparation, time management, neatness, safety, general understanding due to preparation, lab techniques.
(1/8/2011)

Suggestions, comments for: _____ Exp #: _____

_____Well documented experiment

_____ Incomplete documentation of experiment, missing:
_____

_____

product

Analysis.

_____ Definitive, concise.

_____ Incomplete

_____ Need a defining statement on the chemistry of experiment.
_____ missing explanation of data and /or missing data for analysis
_____Need a strong summary conclusion

Other :_____
Abstract.

_____ Definitive, concise.

_____ Incomplete

_____ Not an abstract of experiment
_____ Need a defining statement on the chemistry of experiment.
_____ Give data and analysis.
_____ Need a strong summary conclusion based on objectives
Other :_____
File; suggestionsgradenote2015

8

Code of Academic Integrity

Principle

Absolute integrity is expected of every Cornell student in all academic undertakings. Integrity entails a firm adherence to a set of values, and the values most essential to an academic community are grounded on the concept of honesty with respect to the intellectual efforts of oneself and others. Academic integrity is expected not only in formal coursework situations, but in all University relationships and interactions connected to the educational process, including the use of University resources. While both students and faculty of Cornell assume the responsibility of maintaining and furthering these values, this document is concerned specifically with the conduct of students.

A Cornell student's submission of work for academic credit indicates that the work is the student's own. All outside assistance should be acknowledged, and the student's academic position truthfully reported at all times. In addition, Cornell students have a right to expect academic integrity from each of their peers.

I. GUIDELINES FOR STUDENTS

A. General Responsibilities

1. A student shall in no way misrepresent his or her work.

2. A student shall in no way fraudulently or unfairly advance his or her academic position.

3. A student shall refuse to be a party to another student's failure to maintain academic integrity.

4. A student shall not in any other manner violate the principle of academic integrity.

B. Examples of Violations

The following actions are examples of activities that violate the Code of Academic Integrity and subject their actors to proceedings under the Code. This is not a definitive list.

1. Knowingly representing the work of others as one's own.

2. Using, obtaining, or providing unauthorized assistance on examinations, papers, or any other academic work.

3. Fabricating data in support of laboratory or field work.

4. Forging a signature to certify completion of a course assignment or a recommendation to graduate school.

5. Unfairly advancing one's academic position by hoarding or damaging library materials.

6. Misrepresenting one's academic accomplishments.

C. Specific Guidelines for Courses

1. Examinations. During in-class examinations no student may use, give, or receive any assistance or information not given in the examination or by the proctor. No student may take an examination for another student. Between the time a take-home examination is distributed and the time it is submitted by the student for grading, the student may not consult with any persons other than the course professor and teaching assistants regarding the examination. The student is responsible for understanding the conditions under which the examination will be taken.

2. Course Assignments. Students are encouraged to discuss the content of a course among themselves and to help each other to master it, but no student should receive help in doing a course assignment that is meant to test what he or she can do without help from others. Representing another's work as one's own is plagiarism and a violation of this Code. If materials are taken from published sources the student must clearly and completely cite the source of such materials. Work submitted by a student and used by a faculty member in the determination of a grade in a course may not be submitted by that student in a second course, unless such submission is approved in advance by the faculty member in the second course. If a student is submitting all or part of the same work simultaneously for the determination of a grade in two or more different courses, all faculty members in the courses involved must approve such submissions.

3. Academic Misconduct. A faculty member may impose a grade penalty for any misconduct in the classroom or examination room. Examples of academic misconduct include, but are not limited to, talking during an exam, bringing unauthorized materials into the exam room, and disruptive behavior in the classroom.

a. The faculty member must promptly notify the student of the reason for the imposition of a penalty for academic misconduct and the degree to which his
or her grade will be affected.

b. Academic misconduct is not a violation of academic integrity. The student may, however, seek review by the Academic Integrity Hearing Board on the basis either that the finding of guilt is arbitrary and capricious or that the penalty for academic misconduct is excessive or inappropriate to the circumstances involved. ("Arbitrary and capricious" describes actions which have no sound basis in law, fact, or reason or are grounded solely in bad faith or personal desires. A determination is arbitrary and capricious only if it is one no reasonable mind could reach.)

D. Principles for Computer Use and Network Systems

The use of computers and network systems in no way exempts students from the normal requirements of ethical behavior in the Cornell University community. Use of a computer and network system that is shared by many users imposes certain additional obligations. In particular, data, software and computer capacity have value and must be treated accordingly.

Although some rules are built into computer and network systems, such restrictions cannot limit completely what students can do. In any event students are responsible for their actions whether or not rules are built in, and whether or not they can circumvent them.

Standards of behavior include:

Lab Journal Protocol, Instructions, References

11

The Laboratory Notebook

prepared by Ralph Fleming

Background

Documentation, a system of records, is essential in any laboratory. Documents are a record of what was done, when, how, why, and by who. If a researcher is working on a potential patent or their work is being review by governing agencies, proper documentation is essential.

The Food and Drug Administration, FDA, overseas: the production and distribution of foods and pharmaceuticals, animal testing, clinical trials, medical devices, and over the counter drugs. The policy of the FDA is that if it is not written it was not done. The FDA specifies what shall be recorded and how data shall be recorded in The Code of Federal Regulation, CFR, Title 21, Part 211.194 Laboratory Records. (This part can be found at the fda.gov web site)

The ISO 9000 product quality system, Quality System, ISO 4.2.1 General and Quality Planning 4.2.3 requires documentation as means of ensuring that products conform to specified requirements.

General

The laboratory notebook or journal is the primary form of documentation in the research laboratory. A laboratory notebook is a legal document. It can be used to establish a patent claim, to assign credit for a discovery, to document data for publication, and document historical data. It is critical that the laboratory notebook be used properly and consistently. Since the laboratory notebook has legal significance (grading significance for students), the following formatting rules, guidelines and specifications apply:

Reference: Basic Biotechnology Laboratory Methods in the Regulated Work Place, Madison Area Technical College, Lisa Seidman, NSF ATA grants,

Laboratory Notebook Protocol

The book specification: Composition Book, 9 1/2in. x 7 in. , or 8 ½ in x11in, bound (sewn), ruled pages

- 1st , Enter your name , class , and book volume on the out side cover for identification.
- 2nd, Number every page of book, right side only
- 3rd, Label the top of the first page of the book as : **Table of Content**
- All entries are in chronological order by date
- All entries are in pen: black
- All entries are made direct to book (no scrap paper or other notebooks)

(12)

- Entries are make on right hand pages only, left hand pages are for class notes, calculations, instructions
- State sources for all literature values or outside sources of information.
- Every right page is date when the entry is made
- Every right page has the Experiment # , Experiment name , and date as a header
- Every right page at the end, lower right designates continuance or end
- Every right page is initialed when completed
- The Table of Contents shall reflect all entry pages by Exp. # and Name with page numbers and dates of the work.
- Never rip out any pages
- Cross out, do not erase or white out mistakes. Use a single strike through line and initial the cross out.
- Do not leave blank page or blank areas on a page, draw a diagonal line through the unused portion or pages.
- tables may be drawn in or taped in from a lab manual or spread sheet. Note: proper credit must be given to the author. Include date of entry.
- Hand outs with procedures , charts, reference data, etc. may be taped in with the referenced author , book, etc. and date of entry.
- Spreadsheets may used and be taped in from student developed spread sheets. Note: must have typed on spreadsheet: name, date, citation. Note: proper credit must be given to the author. Include date of entry.
- Use APA format for all citations

Entry Protocol for Organic Chemistry Laboratory Experiments, Che 241, 243

Organic students will obtain from the bookstore:

- **Instructional Supplements for : Organic Chemistry Lab Che-241 or Che-243, BCC,** This will contain a course syllabus and the Experiments.
- **3, bound laboratory notebooks.**

1st, students should familiarize themselves with **Laboratory Note book Protocol** and prepare their books per the **Laboratory Note book Protocol.**

Specific Organic Laboratory Note book Protocols:

- Bound, 8 ½" x 11" sewn composition notebooks either lined or quad ruled are used.
- All entries are made in black ink.
- A Table of Contents on the first page of the notebook lists each experiment by number, name and lab book page number.
- All right side pages in the notebook are numbered consecutively, in the top right corner, starting with page 101. Notebook 2 will start with page 201, etc.
- Right side pages are used for all entries of record; left side pages may be used for rough notes, sketches, etc. The left side pages are **never** used for recording data, procedures or observations.
- Every page is dated in the top right corner. Additional entries made at later dates on the same page are dated at the beginning of the entry.
- Each experiment is titled with the experiment number and the experiment name. Each section of the experiment has a separate header within the experiment
- The order of the section is:
    1. Purpose
    2. General Procedure
    3. Reaction Equation
    4. Preliminary Calculations
    5. Physical Constants Table
    6. Flow Sheet
    7. Data Table
    8. Experimental Calculations
    9. Diagram
    10. Experimental Procedures
    11. Supplements, IR Spectrograms/chromatograms
    12. Analysis
    13. Abstract
- All experimental entries and procedures are made **directly** into the laboratory notebook **as the work is done**.

- Use consecutive, right side pages; do not skip whole pages. However, a page with a title on it, to be completed later, is not a skipped page.
- When an experiment (or an individual section) is continued to another non-consecutive page—use exp. # (section) continued to page... and exp. # (section) continued from page....
- Do not make entries for different experiments on the same page.
- If a mistake is made line through the erroneous entry, leaving it legible, explain briefly and make new entry. **Never erase or tear page out of the notebook.**
- Give sources for all literature values or information cited.

2nd, student should read the experiment to be started completely.

3rd Prepare a list of chemicals to be used in the experiment. All chemicals involved in the experiment are to be listed in your laboratory notebook appendix, starting with the last page and working toward the front.

(In the Appendix of your journal, back of book)

- **Chemical Hazards and Precautions Appendix** (CHP) - all chemicals involved in an experiment are to be listed, with hazards and precautions, in an appendix:

  a. Start the appendix on the last page of the notebook, and work forward, using both right and left side pages.
  b. Number each chemical in the appendix, sequentially for each book; this is the CHP reference number. Example: the first chemical listed in Lab Book 1 would be numbered 1-1.
  c. List the CAS number from the Chemical Identification & Hazard List given to you.
  d. List the NFPA rating as : H-2, F-3, R-1. Notations such as oxy or w are included.
  e. For any chemical with any NFPA rating of 2 or greater, or if the rating sheet indicates - look up and record **all** hazards and precautions.
  f. List spill and waste code letters. Tape comment pages in the back of the lab book. Extra pages are available in lab.
  g. List flash points.
  h. Reference the source of the additional information on the chemicals, i.e. Merck, HCP, Lange, MSDS, NJ fact sheet, etc.
  i. Physical constants may be included for convenience.
  j. A chemical needs to be listed only once; when used in a subsequent experiment, the original entry may be given as the CHP reference number in the experiment Physical Constant Sheet.

CHP APPENDIX

CHP #	Compound	CAS #	NFPA Rating	S	W	Flash Pt.	Reference
11-1	Benzoic Acid	65-85-0	H-2, F-1, R-U	n	n	121°C	MSDS
11-2	Methanol	67-56-1	H-1, F-3, R-0	f	h	12°C	BOTH
11-3	Concentrated Sulfuric Acid	7664-93-9	H-3, F-0, R-2	a	h	208°C	BOTH
11-4	Methyl Benzoate	93-58-3	H-0, F-2, R-0	f	n	83°C	MSDS
11-5	Water	7732-18-5	N/L	N/L	N/L	100°C	NONE
11-6	Diethyl Ether	60-29-7	H-2, F-4, R-1	f	h	-45°C	BOTH
11-7	Sodium Carbonate	497-19-8	H-2, F-0, R-0	n	h	N/L	MSDS
11-8	Sodium Chloride	7647-14-5	H-1, F-0, R-0	n	n	N/L	MSDS
11-9	Anhydrous Magnesium Sulfate	7487-88-9	H-1, F-0, R-0	h	h	N/L	MSDS
11-10	Concentrate Hydrochloric Acid	7647-01-0	H-3, F-0, R-0	a	h	N/L	MSDS

Hazards & Precautions

11-1 **Benzoic Acid:** Warning! Causes eye irritation. May cause irritation to skin and respiratory tract. May form combustible dust concentration in air.

11-2 **Methanol:** Poison! Danger! Vapor harmful. May be fatal or cause blindness if swallowed. Flammable liquid and vapor.

11-3 **Concentrated Sulfuric Acid:** Warning! Causes irritation to skin and eyes and is harmful when ingested.

11-4 **Methyl Benzoate:** Warning! Harmful if swallowed or inhaled. Causes irritation to eyes and skin. Combustible liquid and vapor.

11-5 **Water:** N/L

11-6 **Diethyl Ether:** Danger! Extremely flammable liquid and vapor. Harmful if swallowed, inhaled or absorbed through skin.

11-7 **Sodium Carbonate:** Danger! May cause eye burns. Harmful if swallowed or inhaled. Causes irritation to skin and respiratory tract.

11-8 **Sodium Chloride:** Causes eye irritation.

11-9 **Anhydrous Magnesium Sulfate:** Caution! May be harmful if swallowed.

11-10 **Concentrated Hydrochloric Acid:** Poison! Danger! Corrosive. Liquid and mist cause severe burns to all body tissue. May be fatal if swallowed or inhaled. Inhalation may cause lung damage.

JHS

Dec 1, 2011

EXPERIMENT ENTRIES, The write-up for each experiment includes:

Pre-Laboratory Write-up: This is the **information needed to begin work on the experiment in the lab and must be completed and signed by the instructor or instructional assistant before the student may begin work**. This includes, in the order given:

1. **Purpose**: The reason for doing the experiment.
2. **General Procedure**: A synopsis of the directions from the experiment sheet. This covers all experimental points in brief, sufficient to do the experimental work.
3. **Reaction Equations**: For preparative experiments, equations for all the chemical reactions taking place in the experiment, including main and side reactions. (See Additional Comments). Reactions for purification steps need not be included.
4. **Preliminary Calculations Work Page**: **All** preliminary calculations made involving limiting reagent, theoretical yield, weights, moles, mole ratios, etc., on one page, or pages if necessary.
5. **Physical Constant Sheet**: This form is obtained in the lab and taped into the notebook. All chemicals involved in the experiment are included: CHP numbers and all physical constants necessary in the course of the experiment are given. Limiting reagent, theoretical yield, weights, moles, mole ratios, etc., are included for preparative experiments. (See Additional Comments)
6. **CHP Appendix**: Complete for all chemicals in the experiment (back of book).

The pre-write-up must be written in or word processed, no "cut and pasting" from Experiment Sheets is allowed. The Physical Constant Sheet must be taped in. All pre-write-up sections must precede the first experimental entry.

Laboratory Write-up: The information **obtained and used during the laboratory session. The laboratory write-up must be signed prior to leaving the lab**. This includes, in the order given:

1. **Data Table**: This should be filled in as experimental results are obtained. Each set of data should be identified. DO NOT TYPE DATA TABLE OR RECOPY DATA
2. **Experimental Calculations Work Page**: Includes **all** experimental calculations made during the experimental work and/or involving results, on one page, or pages if necessary.

3. **Flow Sheet**: This is a schematic of the purification procedure which is developed from the general procedures and physical constants for all preparative experiments. (See Additional Comments).
4. **Experimental Procedure**: An exact and detailed record of what is done and observed in the course of the experiment. This is written as the lab work is done. It need not be in sentence form but must be legible and sequential. A diagram of any new apparatus used in the experiment is included. (See Additional Comments).
5. **Spectrographs/chromatograms**: Any such run on the course of the experiment, or given to the student as part of the experiment, are taped in the lab book, properly identified.

Post-Laboratory Write-up: Through CHE 241 the topics to be included in this section will be specified in the experiment sheet. In CHE 243 it will be up to the student to identify and include the necessary information.

The **Analysis** is a statement of the conclusions, which may be arrived at from the results obtained: i.e., a comparison of methods used; yield and purity of a product; etc. Experimental numbers and the corresponding literature values are included in the statement to support the conclusions made.
The analysis only will be graded on the writing. However, in this section as in the entire experiment, the **content** is the major consideration in the grading.
As a guide for the student when writing the analysis, the following information is provided: Each topic paragraph in the analysis should include:
1. a statement of results, including experimental numbers
2. a comparison to literature values or theoretical values
3. a conclusion drawn from the above

The last paragraph is a summary containing the conclusions drawn in the topic paragraphs, without explanation. As for any good writing, it is suggested that the student begin with an outline, to organize the work and make sure nothing is omitted, and write at least one draft before writing the final copy in the lab notebook.

The **Abstract** for you experiment will be a paragraph or two stating the objective, discussing the experiments purpose, the chemistry involved, the procedure and techniques used, the results of the experiment, and a summary or conclusion on the results based on the objective.
An abstract is a brief statement of the essential thoughts of a book, article, body of work, or research.

General Guidelines

EXPERIMENTAL PROCEDURE AND OBSERVATIONS:
Experimental procedure and observations are always written in detail as the work is done. Written procedures and observations should contain sufficient detail that another person could duplicate the work precisely, and know if the results are the same. When work is being recorded properly, a small amount of work is done and then recorded, one step at a time. In the rush to get "work" done the student forgets to write what is done; this is counterproductive. The student will find that by writing as the work is done, over all time is saved, steps will be in sequence and details are not forgotten. When a large amount of work is done before recording, more time is taken, steps are out of sequence and important details are left out. All observations of importance are noted. Used correctly, a laboratory notebook is your memory.

TECHNIQUE EXPERIMENTS AND NEW PROCEDURES:
The first time a new technique or procedure is done, it is written out in detail in the experimental section, including safety precautions, start up, shut down, etc. Thereafter when the procedure is used it is referred to as "standard procedure for ...", noting any variations from the standard, which may affect the results, i.e. "pot" size and heat source for all distillations.

DIAGRAMS:
A diagram of a new apparatus, which is assembled from pieces, is included. Diagrams need not be beautiful, indeed, it is a waste of time to draw a perfect diagram. However, the diagram must be large, and **every** piece must be labeled.

USE OF INSTRUMENTS:
Any time an instrument is used, if there is more than one of that particular instrument, the instrument number is recorded. The first time an instrument is used, directions for use are recorded in detail. However, if an instrument manual is given the student, the pertinent page numbers may be referred to, instead. Thereafter, "standard procedure" is used, noting any items that vary, i.e., "thin film on salt plates" or "sealed cell" for all IR spectra; instrument settings for all melting points, etc. The sequential spectrum number for IR and NMR spectrums is recorded. If an instrument has a log book, it is always signed when the instrument is used.

PREPARATIVE EXPERIMENTS:

The planning of such an experiment is as important as the actual manipulation. A Pre-laboratory write-up is done outside the laboratory period and must be checked by the instructor or instructional assistant before an experiment is begun. If this is not completed before the student comes into the laboratory, valuable lab time is wasted. For the first preparative experiment, a slide-tape presentation is given in independent study, and must be completed as well as a pre-write-up before the experiment is begun.

All preparative experiments consist of two sections, the reaction and then the separation and purification of the product.

1. **Equation For Main Reaction(s):** The first thing to do is choose the reagents and write the balanced equations for the expected reaction.
2. **Side Reactions**: Because of the complexity of most organic molecules, the desired reaction is seldom the only one that actually occurs. Those other reactions are called "side reactions". Write equations for any significant side reactions.
3. **Mole Quantities And Theoretical Yield**: The proper quantities of reagents are usually indicated in the procedure given. The quantity of the most valuable reagent is chosen to give the desired quantity of product. The amounts of other reagents used are then usually greater than that indicated by the main reaction, to allow for loss by side reactions or to act as the solvent. Determine which is the limiting reagent by calculating the number of moles of each reagent used and comparing the ratio of moles used with the theoretical mole ratio indicated by the main equation. Then calculate the number of moles and how many grams of product would be formed from the limiting reagent if there were no side reactions and if the main reaction went to completion (this is the "theoretical yield"). See slide-tape presentation for further details. This information is recorded on the Physical Constant Sheet.
4. **Physical Constants And Solubilities**: A knowledge of the physical properties of the reagents is important in carrying out the reaction, and of the product, side products and secondary products in the isolation of the main product from the reaction mixture. These usually are found in a handbook. Record such physical constants and solubilities as are important for the experiment on the Physical Constant Sheets.
5. **Separation And Purification Of Product**: Frequently, the isolation of the desired product from the other products and excess reagents in the reaction mixture requires more thought and knowledge than does the carrying out of the reaction itself. A procedure for the isolation should be planned before the reaction is run. The steps can be shown in a flow sheet which indicates what happens to each substance present in each step of the isolation. Flow sheets will be provided for the first few experiments, with an increasing number of omissions to be supplied by the student.

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6. **Results**: The product(s) is(are) collected in prelabelled, preweighed bottles (liquids in narrow mouth glass bottles, solids in wide mouth glass bottles) and reweighed to determine the gram yield. Certain tests are run on the product for identification and determination of purity, either during (boiling point) or following (melting point, refractive index, etc.) final purification. Characterization of the product is done by Infrared spectroscopy. NMR standards may be provided. These tests are included in the experimental procedure and the results entered in the Data Table.

Experiments 9, 10, 11, 12

CHEMISTRY 243

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EXPERIMENT IX

CYCLOHEXANONE

REFERENCES: Introduction to Organic Laboratory Techniques, 3rd Ed., Pavia, Lampman and Kriz
Steam Distillation, p. 582-587
NMR Spectroscopy, pp. 713-728

Macroscale and Microscale Organic Experiments, Williamson
Oxidation: Cyclohexanol to Cyclohexanone, pp. 285-287 and 289-290.

OBJECTIVES: The student should be able to:

1. Run a synthetic reaction which requires temperature control.
2. Separate the reaction products by steam distillation.
3. Determine the purity of the product by refractive index and infrared spectroscopy.
4. Calculate the mole, gram, and volume amounts of reactants given the moles of reactants, or the moles of products.
5. Calculate the moles of reactants given the volumes of the reactants and the w:v percentage or molarity of the solution.
6. Identify the product from the NMR spectrum.

INTRODUCTION:

Cyclohexanone is prepared by oxidation from cyclohexanol. The product and any unreacted alcohol are separated from the inorganic reactants and products by direct (in situ) steam distillation of the reaction mixture. The procedure of purification does not separate the alcohol and ketone well; however, the purity of the ketone can be determined by infrared spectroscopy and refractive index.

Since the exact concentration of commercial bleach varies, it is necessary to analyse the reaction mixture for unreacted hypochlorite and to reduce any excess to chloride ion with bisulfite.

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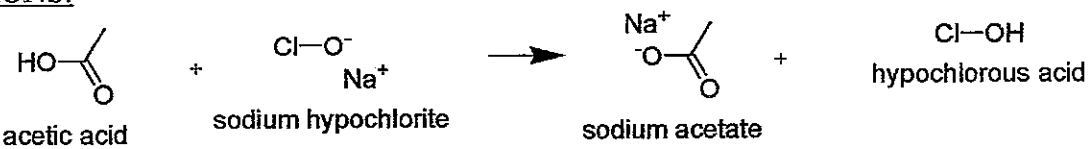
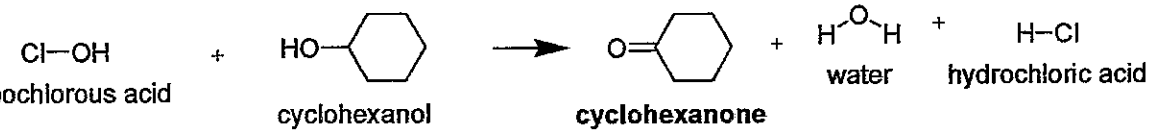
EQUATIONS:

9A 

acetic acid + sodium hypochlorite → sodium acetate + hypochlorous acid

9B 

hypochlorous acid + cyclohexanol → **cyclohexanone** + water + hydrochloric acid

REAGENTS:

Reactants: Cyclohexanol and
Sodium hypochlorite (commercial bleach solution - 5.25% NaClO or 0.75 M)

Initiator: Glacial acetic acid - 6 ml [1]

Calculate the amount of cyclohexanol required to yield 0.100 moles of cyclohexanone, assuming a 100% yield. Use this theoretical amount of cyclohexanol.

Use 155 ml of commercial bleach solution; calculate the mole quantity used. Calculate the mole ratios of the reactants used.

When calculating amounts of reactants, round final masses to 1 decimal place and final volumes to whole numbers.

APPARATUS:

A 300 ml round-bottom flask with thermometer well
A 250 ml dropping funnel (Separatory funnel used for reagent addition)