

MORAVIAN COLLEGE CHEMISTRY DEPARTMENT

Chemistry 212L

Organic Laboratory Techniques

LABORATORY MANUAL

Spring Semester 2006

Dan Libby	213 Collier X-1436	rdlibby@chem.moravian.edu
Carol Baker Libby	213 Collier X-1629	cblibby@chem.moravian.edu

TABLE OF CONTENTS

	Pages
PART I: Introduction:	
Weekly Schedule	2-6
Philosophy	7
Administrative Details	7-9
PART II: Laboratory Rules and Regulations	
A General Daily Routines	10-12
B Safety Regulations	13
C Laboratory Notebook Format	14-16
D Data Binder Organization	17-18
E. Laboratory report Format	19-22
PART III: Explorations	
Exploration 6 Structure and Properties of Stereoisomers: <i>Why are some physical properties the same and some different?</i>	23-26
Exploration 7 Esterification of Vanillin: <i>Do acid and base catalysis yield different results? Why or why not?</i>	27-34
Exploration 8 What is the Effect of Structure on Nucleophilic Substitution Reaction Rates? <i>How can we measure relative reaction rates?</i>	35-40
Exploration 9 Variations on a Theme: <i>How can several different products be synthesized from a common intermediate?</i>	41-47
Exploration 10 How Can a Complex Alkene be Synthesized? <i>A multi-step synthesis of 2-methylheptenes.</i>	48-60
PART IV: Theory	
A Structure and Properties of Stereoisomers	61-71
B General Structure Determination Problems	72-73
C Introduction to the Retrosynthetic Analysis Approach for Designing Organic Syntheses:	74-82

Part I: Introduction:

Weekly Schedule

Week 1	
Laboratory Discussion (Tuesday, January 17)	
Topic:	Chirality and Optical Activity
Assignment:	Lab Manual: Exploration 6: Structure and Properties of Stereoisomers: <i>Why are some physical properties the same and some different?</i> (pp. 23-26) and Part IV A: Stereoisomers sec. A->E (pp. 61-71). Prepare for discussion of questions on pp. 62 - 68. Lehman: Expt. 18 (pp. 136-142) and Oper. 31 Optical Rotation (pp. 693-696) CGWW: Background: pp. 381-396
Laboratory Periods (W-F, January 18-20)	
Activity:	Check-in and Exploration 6: Structure and Properties of Stereoisomers: <i>Why are some physical properties the same and some different?</i> (pp. 23-26)
Assignment:	Exploration 6: Prelab. (p. 23)
Week 2	
Laboratory Discussion (Tuesday, January 24)	
Topic:	Ester Synthesis & Identification of Reaction Products & Chirality and Optical Activity (continued)
Assignment:	Lab Manual: Exploration 7: Esterification of Vanillin: <i>Do acid and base catalysis yield different results? Why or why not?</i> (pp. 27-34) Lehman: Experiment 5 (background on esters and esterification pp. 47-50), Experiment 29 (background on vanillin pp. 224-226) and Experiment 34 (esterification of phenols pp. 266-272).
Laboratory Periods (W-F, January 25- 28)	
Activity:	Exploration 6: Structure and Properties of Stereoisomers: <i>Why are some physical properties the same and some different?</i> (pp. 23-26) & Exploration 7: Esterification of Vanillin: <i>Do acid and base catalysis yield different results? Why or why not?</i> (pp. 27-34)
Assignment:	Exploration 7: Prelab. 1 (p. 27-28)
Week 3	
Laboratory Discussion (Tuesday, January 31)	
Topic:	General Problems in Structure Determination from Spectra
Assignment:	Manual: Part IV B: (pp. 72-73) Propose structures for given data sets.
Laboratory Periods (W-F, February 1-3)	
Activity:	Exploration 7: Esterification of Vanillin: <i>Do acid and base catalysis yield different results? Why or why not?</i> (pp. 27-34)
Assignment:	Exploration 7: Prelab. 2 (p. 28)

Weekly Schedule (continued)

Week 4 Laboratory Discussion (Tuesday, February 7)	
Topic:	Reaction Kinetics - Competing Reactions & General Problems in Structure Determination from Spectra (<i>continued</i>)
Assignment:	Lab Manual: Exploration 8: What is the Effect of Structure on Nucleophilic substitution Reaction Rates? <i>How can we measure relative reaction rates?</i> (pp. 35-40) Part IV B (pp. 72-73) Structure Problems. CGWW: Background on Reaction Kinetics: pp. 381-396
Laboratory Periods (W-F, February 8-10)	
Activity:	Exploration 8: What is the Effect of Structure on Nucleophilic substitution Reaction Rates? <i>How can we measure relative reaction rates?</i> (pp. 35-40)
Assignment:	Exploration 8: Prelab. 1 (pp. 35-36)
Week 5 Laboratory Discussion (Tuesday, February 14)	
Topic:	Nomenclature of Chiral Compounds
Assignment:	Lab Manual: Part IV A sec. F. Nomenclature of Chiral Compounds (p. 70) CGWW: pp. 87-88
Laboratory Periods (W-F, February 15-17)	
Activity:	Exploration 8: What is the Effect of Structure on Nucleophilic substitution Reaction Rates? <i>How can we measure relative reaction rates?</i> (pp. 35-40)
Assignment:	Exploration 8: Prelab. 2 (p. 36)
Due:	Report on Exploration 6
Week 6 Laboratory Discussion (Tuesday, February 21)	
Topic:	Chirality and Optical Activity (<i>continued</i>)
Assignment:	Manual: Part IV A. sec G. Out of Class Application (p. 71)
Laboratory Periods (W-F, February 22-24)	
Activity:	Exploration 8: What is the Effect of Structure on Nucleophilic substitution Reaction Rates? <i>How can we measure relative reaction rates?</i> (pp. 35-40)
Assignment:	Exploration 8: Prelab. 3 (p. 36)

Week 7

Laboratory Discussion

(Tuesday, February 28)

Topic: Divergent Multistep Syntheses

Assignment: Lab Manual: Exploration 9: Variations on a theme: *How can several different products be synthesized from a common intermediate?* (pp. 41-47)

Lehman: Experiment 56 (pp. 449-456), and Minilab 33 (pp. 511-513)

Laboratory Periods

(W-F, March 1-3)

Activity: Exploration 9: Variations on a theme: *How can several different products be synthesized from a common intermediate?* (pp. 41-47)

Assignment: Exploration 9: Activity A: Prelab. (p. 41)

Week 8

Laboratory Discussion

(Tuesday, March 14)

Topic: Introduction to the Retrosynthetic Analysis – Synthesis of Alcohols

Assignment: CGWW: pp. 771-774, 788

Lehman: pp. 244-249 (Grignard Reaction)

Manual: Part IV C sections A. Intro (p. 74)

Laboratory Periods

(W-F March 15-17)

Activity: Exploration 9: Variations on a theme: *How can several different products be synthesized from a common intermediate?* (pp. 41-47)

Assignment: Exploration 9: Activity B: Prelab. (p. 42)

Due: Report on Exploration 7

Weeks 9

Laboratory Discussion

(Tuesday, March 21)

Topic: Retrosynthetic Approach to Alkene Synthesis: FGI dehydration

Assignment: Lehman: Experiment 21 (pp. 156-165)

CGWW: pp. 482-484 & 487-490

Manual: Part IV C sec. C. 1. Alkene Synthesis by Dehydration of Alcohols (p. 78)

Laboratory Periods

(W-F, March 22-24)

Activity: Exploration 9: Variations on a theme: *How can several different products be synthesized from a common intermediate?* (pp. 41-47)

Assignment: Exploration 9: Activity C: Prelab. 1 (p. 43)

Weekly Schedule (continued)**Week 10****Laboratory Discussion**

(Tuesday, March 28)

Topic: Use of SciFinder Scholar for Literature Searches
Meet in Reeves Library with Librarian Wendy Juniper

Assignment: CHEM 212 Library Project Handout

Laboratory Periods

(F-W-Th, March 31-April 5 & 6) – No Lab on Wed. & Thurs., March 29 & 30

Activity: Exploration 10: How can a Complex Alkene be Synthesized?
A multi-step synthesis of 2-methylheptenes. (48-60)

Assignment: Exploration 10: Activity A - Prelab. 1 (pp. 48-49)

Due: Report on Exploration 8

NOTE: Although there will be no lab meetings on W & Th, the reports are due as follows:

W & Th Mar 29 & 30 By 5:00 PM F in lab sec.

Week 11**Laboratory Discussion**

(Tuesday, April 4)

Topic: No Laboratory Discussion – Martin Luther King Day Observance Activities

Laboratory Periods

(F-W-Th April 7-12-13)

Activity: Exploration 10: How can a Complex Alkene be Synthesized?
A multi-step synthesis of 2-methylheptenes. (48-60)

Assignment: Exploration 10: Activity A - Prelab. 2 (p. 49)

Week 12**Laboratory Discussion**

(Tuesday, April 11)

Topic: Retrosynthetic “FGI oxidation” for Synthesis of Ketones

Assignment: Manual: Part IV C sec. D. Oxidation of Alcohols to Ketones (pp. 80-81)

Laboratory Periods

No Lab Friday, April 14 – Easter Break

Week 13

Laboratory Discussion

(Tuesday, April 18)

Topic:

Alcohol Synthesis
and
Multiple Disconnections in Organic Syntheses

Assignment: Lehman: pp. 244-249 (Grignard Reaction)

Manual: Part IV C sec. A->D. Retrosynthetic Analysis (pp. 74-82)

Laboratory Periods

(W-F April 19-21)

Activity: Exploration 10:How can a Complex Alkene be Synthesized?
A multi-step synthesis of 2-methylheptenes. (48-60)
& Check Out

Assignment: Exploration 10 Activity B - Prelab. (p. 55)

Due: Report on Exploration 9

Week 14

Laboratory Discussion

(Tuesday, April 25)

Topic: General Class Discussion

Assignment: Current Class Group Activity

Laboratory Periods

(W-F April 19-21)

Activity: Exploration 10:How can a Complex Alkene be Synthesized?
A multi-step synthesis of 2-methylheptenes. (48-60)
& Check Out

Assignment: Complete Exploration 10 Activity B

Due: Lab report for Exploration 10 (Part III, pp. 57-59) and lab notebook & data binder (with all notebook checklists, data and returned lab reports behind appropriate tabs).

Due by 5:00 PM on the day after your last lab period.

Philosophy

(See also Lehman pp. 2-9)

The laboratory part of Chemistry 211-212 is divided into two parts with different emphases. First semester, Chemistry 211 laboratory was an exploratory introduction to the major standard techniques used by organic chemists. Second semester, Chemistry 212 laboratory utilizes these techniques and a few new ones to synthesize organic molecules and investigate their structures and behavior.

This semester you will be working on more complex problems than you did in CH 211, so again, **IT IS ABSOLUTELY ESSENTIAL THAT YOU PREPARE FOR EACH EXPLORATION AHEAD OF TIME.** *Without a general understanding of the overall goals of each exploration, and a plan for carrying out the procedure, you will have great difficulty in successfully completing the work presented to you.* Again this semester there will be discussions and assignments to help you operate efficiently in the laboratory. There will be background reading assignments to assist you in designing your experimental plans for each exploration, laboratory discussions on Tuesdays, written prelab assignments for each lab period, and a daily notebook check and quiz. (See Weekly Schedule on pp. 2-6) Again, you should do all of the assigned reading and preliminary preparation for the week's activities before the Tuesday discussion (9:10-10:00 AM). Then bring any questions to the Tuesday discussion so that we can solve problems before they arise. The written prelab assignments are due when you come to lab and again you will have a quiz while your pre-lab assignment is checked by your instructor before you are allowed to start work on the day's exploration.

Administrative Details.

Required Materials; (Same as for CHEM 211L)

- Lab Text: *Operational Organic Chemistry: A Problem-Solving Approach to the Laboratory Course*, by John W. Lehman, Prentice-Hall 1999 (Lehman)
- Lab Notebook: Must have a hard cover and be permanently bound (not spiral), must also fit in the inside pocket of your data binder. You may continue in your notebook from CHEM 211 (See p. 8).
- Data Binder: A 1.5-inch three-ring loose-leaf binder with a hard cover and at least 6 tab dividers and an inside pocket that will accommodate your lab notebook. You may continue in your data binder from CHEM 211.
- Lab Goggles These must fit tightly against your face.

Grades

In general, satisfactory preparation for each lab period, completion of all work, laboratory notebook records and laboratory reports including answers to all prelab and postlab questions will earn a grade of B for the lab. Higher grades will be given for demonstration of excellent understanding of the concepts, preparation for the lab periods, performance in the lab, well organized and thorough lab records and well-written lab reports.

Your grade for the laboratory portion of the course will be calculated as indicated below:

- 10% Attendance in the Tuesday discussion and laboratory periods
- 10% Attendance in the laboratory
- 10% The quality and completeness of the written prelab assignments
- 10% Apparent preparation for each laboratory period
- 10% Evidence of increasing skill and initiative in the laboratory
- 25% The quality and completeness of the laboratory notebook and data binder
- 25% The quality and completeness of the laboratory reports

Attendance

Students are required to attend all laboratory discussions (Tues. 9:10-10:00) and all scheduled laboratory periods (See the Weekly Schedule on pp. 2-6) unless excused due to a valid medical excuse (verified by a physician, the Health Center or Dean of Students' office) or other accepted prior excuse. Make-ups or grade adjustments for excused absences will be arranged. Grades for work missed due to unexcused absences will be zero.

Note: Work conflicts or travel schedules for vacations are not acceptable excuses for missed discussion or lab periods.

Lab Records

Notebook

As in CHEM 211L, all notebooks must be permanently bound hard cover record books not spiral bound books. Your records should be permanent; so all entries must be made in non-erasable ink. You may continue with the same notebook used in CHEM 211L, but you must be sure the laboratory section designations on the front and bottom page edges are correct for this semester. Whether you use your CHEM 211L notebook or not, you should have the material from CHEM 211L available for information on lab techniques.

Data Binders

Again this semester, your data binder will provide you with a convenient place to keep the spectra, chromatographs and other sheets of data generated by your lab work. If it has sufficient space, you may continue to use the your binder from CHEM 211L. Just add additional tabs for this semester's explorations. Whether you use your CHEM 211L binder or not, you should have the material from CHEM 211L available for information on lab techniques.

Laboratory Reports

Once your laboratory work on each exploration is complete you will summarize and interpret your results in a formal laboratory report, which you will prepare using a word processing program. The Weekly Schedule (pp. 2-6) lists specific due dates for your laboratory reports. The format for lab reports is provided in Part II - E (pp. 19-20) and specific instructions for each report are provided in each exploration.

Preparation for Laboratory Discussions

Similarly to last semester, the Tuesday class meeting will focus on laboratory explorations and related materials. The Weekly Schedule (pp. 2-6) provides references to relevant sections in this manual, and the laboratory (Lehman) and class (CGWW) textbooks that discuss the background theories and techniques you will be using in your laboratory work. You should be familiar with the material in these assignments before the Tuesday lab discussion.

Preparation for Laboratory Periods and Evaluation of Laboratory Notebooks

The Weekly Schedule (pp. 2-6) also includes the specific assignments for your lab periods including weekly written prelab assignments and pre- and post lab notebook checks.

Collaboration and Academic Honesty

Collaboration among students in the laboratory and on analysis of data is generally encouraged and required for some explorations. However, the final version of all written work (**pre-lab assignments and quizzes, and laboratory reports**) submitted for evaluation must be individually prepared in your own words after consultation with other students. To be fair to all students in the course and to assure maximum learning for each student, we follow all the guidelines for academic honesty spelled out in the *Moravian College Student Handbook 2005-2006* (pages 54-59). Particularly relevant passages are excerpted on the following page.

Academic Honesty at Moravian College

Academic integrity is the foundation on which learning at Moravian College is built. Moravian College expects its students to perform their academic work honestly and *fairly*. In addition, a Moravian student should neither hinder nor unfairly assist the efforts of other students to complete their work successfully. The College's expectations and the consequences of failure to meet those expectations are outlined below.

In an academic community, students are encouraged to help one another learn. In fact, because no two students learn in exactly the same way or take exactly the same things away from a lecture, the College encourages students to *study* together. The boundaries on what is or is not acceptable work *may* not always be clear; thus, if at any point in academic work at Moravian the student is uncertain about his or her responsibility as a scholar or about the propriety of a particular action, the instructor should be consulted. The list below is not to be considered complete but rather covers the most common areas of concern. In general, students should be guided by the following principles.

Plagiarism

A major form of academic dishonesty is plagiarism, which the College defines as the use, whether deliberate or not, of any outside source without proper acknowledgement. While the work of others often constitutes a necessary resource for academic research, such work must be properly used and credited to the original author. This principle applies to professional scholars as well as to students. An "outside source" is any work (published or unpublished) composed, written, or created by any person other than the student who submitted the work. . . . All work that students submit or present as part of course assignments or requirements must be their original work unless otherwise expressly permitted by the instructor. . . . When students use the specific thoughts, ideas, writings, or expressions of others, they must accompany each instance of use with some form of attribution to the source. Direct quotes from any source (including the Internet) must be placed in quotation marks (or otherwise marked appropriately) and accompanied by proper citation, following the preferred bibliographic conventions of the department or instructor. . . . Student ignorance of bibliographic convention and citation procedures is not a valid excuse for having committed plagiarism. . . . A student may not present oral or written reports written by others as his or her own work. . . . You may not use writings or research obtained from any other student previously or currently enrolled at Moravian or elsewhere or from the files of any student organization (such as fraternity or sorority files) unless expressly permitted to do so by the instructor. . . . Students must keep all notes, drafts, and materials used in preparing assignments until a final course grade is given. . . . All such materials must be available for inspection by the instructor at any time.

Cheating

Students may not submit homework, computer solutions, lab reports or any other coursework prepared by, copied from, or dictated by others. If the student is employing the services of a tutor (whether from the College community or elsewhere), the tutor may not prepare the student's work for class. . . .

False Testimony

Students may not submit or present a falsified excuse for an absence from an examination test, quiz, or other course requirement either directly or through another source.

Students may not falsify laboratory results, research data, or results. They may not invent bibliographical entries for research papers, websites, or handouts.

I. Introduction

10

They may not falsify information about the date of submission for any coursework.