

Table of Contents

- ❑ Schedule of Experiments
- ❑ Lab Requirements
 - Supplies
 - Preparation for Lab
 - Grade
 - Course Philosophy
 - Lab Reports, Example Experimental Procedure
- ❑ Helpful Tips for Success
- ❑ Laboratory Safety

Schedule of Experiments

Fall 2008

Sept. 3/4 Introduction/Methylation of 5-Methyl-salicylic Acid

Techniques: Advanced synthetic organic techniques: conducting reactions under anaerobic conditions with dangerous chemicals

- . *work in groups of 2-3*
- . *lab notebook*

Sept. 10/11 Reduction of L-Proline

Techniques: Advanced synthetic organic techniques: conducting reactions under anaerobic conditions with dangerous chemicals; product isolation and identification

- . *work in groups of 2-3*
- . *lab notebook*

Sept. 17/18 Amide Synthesis

Techniques: Advanced synthetic organic techniques: conducting reactions under anaerobic reactions with dangerous chemicals

- . *work in groups of 2-3*
- . *lab notebook*

Sept. 24/25 Amide Purification

Techniques: Isolation and purification of organic products with flash column chromatography; product analysis including COSY 2D NMR experiment

- . *work in groups of 2-3*
- . *lab notebook*

Oct. 1/2 Methyl Ether Synthesis

Techniques: Advanced synthetic organic techniques: conducting reactions under anaerobic reactions with dangerous chemicals

- . *work in groups of 2-3*
- . *lab notebook*

Oct. 8/9 Methyl Ether Purification

Techniques: Isolation and purification of organic products with flash column chromatography; product analysis

- . *work in groups of 2-3*
- . *lab notebook*

Requirements

Fall 2008

Lecture: Wednesdays 1:30-2:30 PM PSB 229

Laboratory: Thursdays 1-5/6 pm PSB 274

Course website: <http://www.brynmawr.edu/chemistry/malachowski/ResMethod251/ResMethod251intro.html>

Instructors: **Prof. Bill Malachowski** **Prof. Jonas Goldsmith**
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assisted by: **Rebecca Rothstein, BMC '09** **Sarah Miller, BMC '09**

Final Grade: Synthesis Portion: 45%

- Synthetic project 30%
 - notebook evaluations 10%
 - yields 10%
 - final report 10%
- Final exam 15%

Physical Chemistry Portion: 45% (breakdown described later)

Attendance and summary of research colloquia 10%

Lab Manual: The manual will be dispensed in pieces as we begin each new experiment. You may want to purchase a three-ring notebook to assemble the complete manual.

Required equipment: Goggles
Bound notebook (i.e. composition book)
Sharpie-type marker for labeling glassware
Apron or lab coat (optional)
Gloves (available in lab)

Lab Notebook: You are required to keep a lab notebook. A bound composition book is perfect for the job; spiral binders or notebooks with tear-out sheets or carbon-copy pages are not. You will use this notebook to record all your data during the experiment. You should write a brief procedure for the day's experiment before you come to lab. Your lab notebook will be evaluated each week. Notebooks will be collected by 5 PM on each Friday, graded each weekend and returned on Mon. by 6 PM.

Preparation for Lab: BEFORE COMING TO LAB, you should prepare yourself to undertake the week's experiments. Writing a procedure in your lab notebook is one device to help you prepare yourself. To further prepare you for lab action, you may be asked to answer several short questions prior to arrival. LAB NOTEBOOKS MAY BE CHECKED BEFORE THE LAB PERIOD.

Final Lab Report: Details of the final lab report are given below. A rough draft of the introduction section will be due on Fri. Sept. 12 using the *Journal of Organic Chemistry* template. A second rough draft containing the introduction and a preliminary results and experimental section will be due on Fri. Sept. 26. A third rough draft will be due on Fri. Oct. 10. The final lab report will be due in class on Wed. Oct. 22.

Final Exam: There will be a 1.5 hr. take-home final exam. Exams from previous years are available on the course website.

Colloquium Summaries: You are required to attend at least four colloquia (research seminar series) over the course of the semester and to prepare a summary (one page or less) for at least three of these. Additional summaries and attendance will be counted as extra credit. The Fall 2008 seminar series schedule can be found at <http://www.brynmawr.edu/chemistry/colloq.html>.

The summaries should include: a) a brief statement of the purpose, goal or objective of the speaker's research work; b) a description of at least one method of analysis used in the work, including a description of the fundamental basis for the method and an example of the resultant data interpretation.

Statement of Philosophy of the Course

As Research Methodology is an upper level course, you may think of it as the next step in your development as a chemist. Taking this perspective, several goals have been set for you to achieve over the course of the semester.

The first goal is to develop your skills as an independent thinker in the laboratory. The procedures in this course are less specific than what you have previously encountered. Thus, YOU will learn to make decisions on what size and type of equipment you should use, etc and you should use the two years of chemistry laboratory experience to help you make these decisions in the lab. Of course you are welcome to ask for advice or confirmation from Prof. Malachowski, Prof. Goldsmith and the TA's as they are here to assist you in this process.

The second goal, which is closely related to the first, is to further prepare you for doing research, either at Bryn Mawr or elsewhere in your career. A practicing scientist needs to keep an accurate notebook to verify scientific claims and for the awarding of patents. Therefore, you are required to keep a notebook for this class in a manner similar to the general or organic chemistry lab. One format you may choose to use is to start by stating the objective of the experiment. You can follow this by either writing the relevant reaction(s) or by briefly writing down the procedure. Data tables should also be set up before entering lab. After lab you may want to record your thoughts as you analyze your data and draw conclusions. Above all, your notebook should be legible and full of observations (physical chemistry deals with a lot of colors which are important to record!).

The last point to make about this course is that it is not only a combined course in Organic and Physical Chemistry, but also an Instrumental Analysis course. Some of the instruments you may have already used, but you will also be learning about many other techniques as well.

Have fun with the semester and enjoy the excitement of advanced experimentation. Please come to lab prepared to work and to expand your knowledge of chemistry. Good Luck!

LAB REPORTS

The purpose of writing lab reports in this course is two-fold. First, it is meant to further develop your writing skills in a scientific context. The goal here is to write more in the style found in the scientific journals. The second purpose is to give you the opportunity to show us what you have learned in lab in terms of theory and techniques.

You should use a Note template from the *Journal of Organic Chemistry*, which can be downloaded at the following website:

<http://pubs.acs.org/paragonplus/submission/joceaah/index.html>

It would also be very useful to use other literature articles as a guide for the proper format.

A complete lab report has the following elements:

Introduction: What is the goal? Why is it of interest to us? What are the scientific objectives of the experiment? What related work has already been reported in the literature? How does this work differ? Attention should be given to both chemical and analytical aspects of the experiment. Chemical aspects include relevant chemical equations and special chemistry/details of a synthesis. Analytical aspects should include a **BRIEF** but complete discussion of **NEW** spectroscopic techniques employed in each experiment including the theory behind the technique and the useful information obtained from using this technique.

Results and Discussion: Summarize your results in an **ORGANIZED** fashion and comment on them. What contributed to poor yields or impure products? What techniques or procedures were essential for success? Comparisons to literature procedures can be made. Tables can be used to present data. Be sure to label and include units. Remember that you are just describing your results not trying to interpret them at this point.

Experimental: A brief summary of the procedure, written so that someone "trained in the art" could reproduce your results. In other words, written for someone who is knowledgeable in basic chemical techniques and glassware/equipment used in syntheses. Compound characterization and purity information needs to be detailed here. This section also includes the description of your observations.

References: A list of sources used for background information, experimental procedure, and literature values should be presented. You may find that you are just referencing the literature article used in the course; that's OK.

Other details:

1. Lab reports are to be typewritten (or word processed) and use ChemDraw structures with the ACS Document 1996 format (see selection under File tab).

2. The standard verb tense for scientific writing is the **passive past tense**. For example: "The solid was collected by vacuum filtration," not "The solid is collected by vacuum filtration." *This verb tense mostly applies to the experimental section of the report.*
3. The entire report (including procedure) should be in **PARAGRAPH FORM**, not in outline form.
4. Use abbreviations whenever appropriate. A list of commonly accepted abbreviations in organic chemistry can be found at the *Journal of Organic Chemistry* web site in the 'Info for Authors' section. In particular see page 12 and 13 of the following author guide: http://pubs.acs.org/paragonplus/submission/joceah/joceah_authguide.pdf
If an uncommon abbreviation is used, then you must define it the first time that it is used.
5. Be specific!! "The carboxylic acid" does not correctly identify the material. Use compound numbers to refer to specific structures; e.g. "...carboxylic acid **2** was heated...".
6. Your lab report should be written in *your own words*!

Helpful Tips for Success

Fall 2008

Here are some helpful tips regarding some of the procedures in the lab.

1. *Cleanliness and organization are essential for maximum productivity.*
 - a) Please keep all common lab areas clean and neat. If you make a mess, please clean it up within 1 hour. Others do not know what you have spilled and therefore this mess represents a dangerous safety hazard.
 - b) Glassware cleaning procedure:
 - (1) Soak and scrub dirty glassware in soap water.
 - (2) Wash off soap with tap water.
 - (3) Wash with deionized water
 - (4) Wash with acetone and leave dry. Acetone washings should be collected and put in the non-halogenated organic waste jug. Acetone should not go down the drain. The Environmental Protection Agency will fine people who dispose of chemicals down drains.
2. *Listen to your instructors, they would never intentionally lead you astray.*

Dress You should dress appropriately for lab. On days that we are doing synthesis, it is strongly suggested to wear older clothes as we will be working with acids/bases and solvents that can harm clothing. Please observe the following guidelines when dressing for lab.

1. NO sandals or open-toed shoes. Socks with sandals are NOT acceptable. Sneakers are probably your best choice.
2. No shorts, really short skirts or sleeveless shirts.
3. Long hair must be tied back off the face.
4. Avoid jewelry if possible.

Be prepared to return to your dorm if you are dressed inappropriately.

If you have an apron or lab coat, please bring it to wear during lab if you desire.

Eye Protection Some sort of eye protection is required in lab at all times. However, you do not have necessarily to wear goggles all the time either.

Goggles Goggles are required on days that we are doing synthesis. Please bring yours and leave them in your lab drawer.

If you are a contact lens wearer, it is OK to wear them to lab.

Gloves The chemicals used in lab are dangerous and consequently you will often need to wear gloves. Nitrile gloves will be available for your use in the lab. Consider the gloves to be a disposable layer of skin. If you get chemical or solvent on the gloves, remove and discard them immediately. Get a fresh pair of gloves.

A few words about chemical hygiene:

Remember that you are wearing gloves to protect *you* from absorbing or ingesting the chemicals that we are working with in lab. However, if you pick up your pen to write in your notebook while wearing the gloves on which you just spilled some solvent, you have really defeated the purpose of the gloves. Whatever was on your gloves is now on your pen which you will take with you to your next class or to use to do your homework. So think about what you are touching when you have your gloves on.

This also applies to opening doors with your gloves on your way to the instrument room to take a spectrum.

*Waste
Disposal*

Many of the chemicals we will be using cannot be disposed of down the sink. Waste Disposal instructions are clearly written as part of the procedures found in your manual. Please read them carefully before coming to class.

Read labels on waste jugs carefully as well.

As you probably know by now, glass cannot go in the regular trash. There are special cardboard glass disposal boxes located in the lab. Be sure to place broken glass in these containers.

*Safety
Equipment*

When you get into lab, please locate the following safety equipment in case there is an emergency.

1. fire extinguishers
2. eye washes
3. safety shower
4. first aid station
5. fire alarm
6. emergency phone

In case of an actual emergency, use the yellow emergency phone in the hall or call public safety at ext. 7911 or 7310.

If you have a medical condition that we should know about, please talk to us as early as possible.