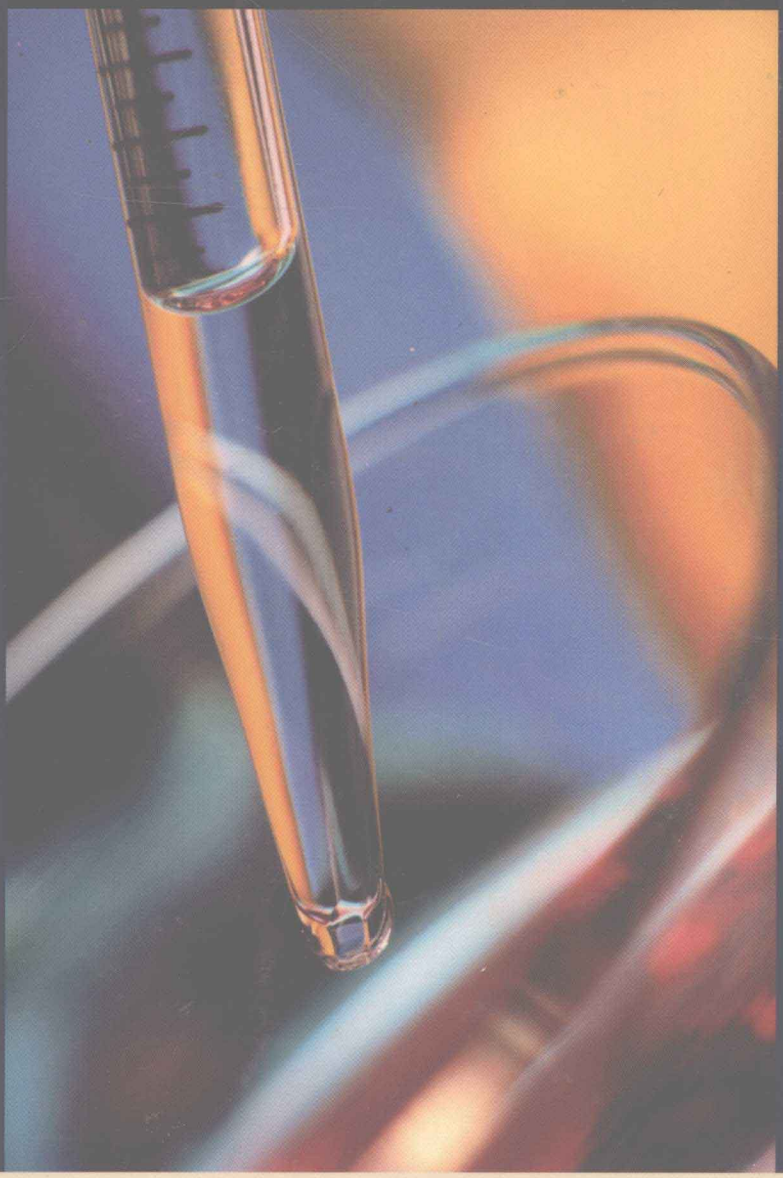
# EXPERIMENTAL ORGANIC CHEMISTRY

## A SYNTHETIC & MECHANISTIC PERSPECTIVE



RALPH NICHOLAS SALVATORE / KYUNG WOON JUNG

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**Ralph Nicholas Salvatore**

**Kyung Woon Jung**

**University of South Florida**

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Finally, we dedicate this book to our family and special friends whom we wish to thank for their encouragement, support, and patience.

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# Chapter 1

# General Guidelines for Organic Chemistry Laboratories I & II

## Post-Lab Report

It is strongly encouraged that one should try to make all reports written reasonably and scientifically. No emotional essays will be accepted.

### I. Introduction

State briefly the purpose of the experiment that you performed.

### II. Theoretical Background

With use of any organic textbooks or referenced books, one should include any necessary theoretical background such as reaction mechanisms and physical properties. Point out only salient features (*i.e.,* side reactions), which one should be concerned about in an effort to obtain the best experimental result.

### III. Data & Results

Re-organize the raw data and post it in the report.

### IV. Discussion

You should evaluate your data and results, and make comments on how good and how bad they were. Explain the reasons and causes of the results, and how one can seek a better protocol based on appropriate approaches, encompassing logical rationale and theoretical background. Any supporting evidence or precedents should be mentioned in your own style and the references that you use should be cited in the References section. Any reasonable discussion is welcome, for example, better experimental design and applications.

### V. References

The aforementioned sections should be based on known knowledge or your own reasoning. If you adopt any material from books and journals, you should cite them therein appropriately. Information obtained from the internet must be cited as well.

## Lab Reports

Each of the experiments performed will involve the write-up of a formal report. It should present your results and conclusions clearly in an organized fashion with the purpose of convincing the reader you have achieved and understood the goal of each experiment. Its success will depend partly on the quality of the information obtained, and largely on the organization and interpretation of the information. A narrative or writing of your "memoirs" will not be acceptable. The report should be neat, concise, and clearly convey your point. Some comments on a suitable organization of your report are discussed below.

- All laboratory reports should be written neatly in blue or black ink. Typing is also acceptable. If a mistake is made, the incorrect entry should have one line drawn through it. Do not scribble over incorrect entries. White out should never be used in scientific reports.

- All portions of the lab report should be labeled clearly. For example: Introduction, Results, Discussion, etc. Refer to the post-lab report guidelines for appropriate sections.

- The results of the experiment should be clearly and concisely discussed. Focus on interpretation, rather than a rambling plot approach. The report should include a certain amount of scientific information deemed relevant. However, a report with large amount of writing <u>does not</u> guarantee a good grade. Organization of raw data in table form may help.

- Mechanistic interpretation of each organic reaction should be discussed in the report. You should push the arrows in the proper fashion. Consult the literature for help with this.

- Any possible side reactions that may occur during the experiment should be discussed and shown through realistic equations.

- Keep in mind the following points to include when organizing your report: Did you identify the compound if it was an unknown? Summarize the evidence of the identity. Evidence of purity and physical properties, including literature references. Spectroscopic properties and interpretation, including literature references. Chemical behavior and interpretation, including chemical equations. Properties of the derivative if one was made, including literature references.

- You should properly reference all literature used for each experiment in the proper fashion. If textbooks are used, you should cite them accordingly with the author's name, title, publisher, and year. If the world wide web is used, provide the appropriate web address.

## Laboratory Notebook

- Use a bound notebook preferably pre-numbered by the manufacturer. A notebook containing removable duplicate pages with carbon copies is optional. No particular format for writing in the lab notebook will be required. However, keep the following points in mind:

- Each page should include a date, and a title of the experiment performed.

- You should record all data and observations while the experiment is in progress. Pre-lab write-ups which should include an experimental procedure, chemical and physical data on chemicals used, preliminary data, such as chemical structures, chemical reactions, hazardous and toxicity data, etc. should all be written before the lab. For properties of chemicals, see The Merck Index, Sigma or Aldrich Catalogue, CRC Handbook, etc.

- Data and observations should not be changed unless an error is made or a step is repeated. The original value should be crossed out legibly and rewritten.

- Balanced chemical equations, principal side reactions, and mechanisms of organic reactions will be included in the formal report. You do not have to include them in your notebook. For mechanistic information, see any standard Organic Chemistry Laboratory Textbooks.

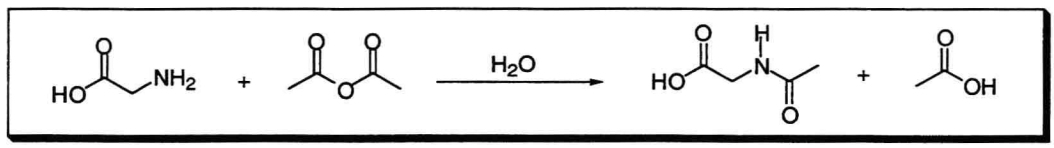
- Draw conclusions from the observations made. This part, of course, can be written after leaving the laboratory. This includes percent yields. See the sample calculation.

# Sample Percent Yield Calculation

A **percent yield** is simply the percent of the theoretical amount of product obtained in a reaction. You must be familiar with this calculation for all synthetic reactions in this course.

$$\% \text{ yield} = \frac{\text{actual}}{\text{theoretical}} \times 100$$

**EXAMPLE**: In the following synthesis, 1.0 g of glycine was acetylated using 2.5 g of acetic anhydride. At the end of the experiment, 1.38 g of acetylglycine had been isolated. Calculate the percent yield of acetylglycine obtained from the following reaction.




1) Calculate all molecular weights.

2) Calculate the number of moles of each reagent involved in the reaction. Do not consider catalytic amounts of reagents or solvent.

| | Glycine | Acetic Anhydride | Acetylglycine |
|---|---|---|---|
| MW: | 75.1 g/mol | 102 g/mol | 117 g/mol |
| weight: | 1.0 g | 2.5 g | ? |
| moles: | 0.0133 | 0.0245 | ? |

3) Determine the **limiting reagent** (the reagent present in the least amount). This information comes from the balanced chemical equation. This equation is balanced as it stands, so the stoichiometry of the reactants is 1:1. Therefore, in this particular reaction, glycine is the limiting reagent, while the acetic acid is present in excess.

4) Determine the theoretical number of moles of product possible. In this case, 0.0133 mole of glycine is the maximum yield. This value is the same as the limiting reagent value in moles.

5) Convert the theoretical yield to grams.

0.0133 mole of glycine x 117 g/mol =1.56 g (theoretical yield)

6) Use the percent yield equation above.

(1.38 g/1.56 g) x 100 = 88%

# LABORATORY SAFETY RULES

**EYE PROTECTION**: Splash goggles (American Optical 4848, Cesco 522-C, or departmentally approved equivalent) must be worn over the eyes at all time in the laboratory, regardless of what is being done. Contact lenses should not be worn during the lab period.

**FOOTWEAR**: Shoes must be worn at all times during the laboratory. The feet must be adequately covered (the foot must be totally covered up to the ankle). Therefore sandals and open-toed shoes are not acceptable.

**CLOTHING**: Clothing must be worn which covers the same parts of the body that are covered by a short sleeve full length lab coat (coverage must be down below the knee and down the arm to a point halfway from the shoulder to the elbow). Therefore, tank tops, halters, shorts, cutoffs, etc. are not acceptable. Lab coats and aprons may be worn but will not be accepted as a substitute for proper clothing.

**HAIR**: If hair is long enough to interfere with the experiment, it must be tied back.

**FOOD:** Eating or drinking in the laboratories is prohibited.

**SMOKING:** Smoking in or near the laboratories is prohibited at all times.

**OPEN FLAMES**: Open flames of any type are prohibited in the laboratory, unless specific permission is granted to use them.

**SCHEDULE:** Students will work only during their scheduled laboratory period and never alone or unsupervised.

**EMERGENCY EQUIPMENT:** Know the location and use of all safety equipment and exits.

**CHEMICALS:** A) Never taste any chemical B) Never pipet with your mouth C) If you spill chemicals on your hands or body, immediately flush liberally with water. Get further directions from your instructor D) Use chemicals that generate harmful vapors in the hood only E) Return reagent bottles to their place after using. Never pour unused chemicals back into reagent bottles.

**WASTE DISPOSAL:** Chemicals and used materials should be discarded in specified containers. When in doubt check with your instructor.

**ACCIDENTS:** Report all accidents however minor to your instructor immediately.

**WORK SPACE:** Keep your working space neat at all times and clean up when you leave for the day. Return equipment to its proper place.

# Chapter 2

# Organic Chemistry Laboratory I Experiments

## Organic Chemistry Laboratory I Syllabus

This course is designed to provide students with the fundamental techniques of organic chemistry including methods of isolation, purification, and structural identification with applications to synthetic and mechanistic problems. This course will focus on fundamental reactions and techniques applicable to various fields of organic chemistry. Students are advised to understand, prior to coming to lab, what they will perform in practice, and they are also strongly encouraged to use a problem solving approach during the class as well as while writing post lab reports. Scientific organization and writing will be other goals of this course.

### Schedule of Experiments

| | |
|---|---|
| **Lab Meeting # 1** | Check-in and Safety lecture. |
| **Lab Meeting # 2** | Exp. 1: Simple and Fractional Distillation of a Binary Mixture. |
| **Lab Meeting # 3** | Exp. 2: Acid-Base Extraction: Separation of an Organic Acid, a Base, and a Neutral Substance. |
| **Lab Meeting # 4** | Exp. 3: Chromatography-Identification of the Composition of an Unknown Analgesic Tablet and Isolation of β-Carotene from Spinach Leaves. |
| **Lab Meeting # 5** | Exp. 4: Chirality-Isolation of Limonene from Citrus Fruits. |
| **Lab Meeting # 6** | Exp. 5: Isolation of Trimyristin from Nutmeg. |
| **Lab Meeting # 7** | Exp. 6: Preparation of Myristic Acid from Trimyristin by Hydrolysis. |