Laboratory Notebooks

I. Introduction

Purpose

Laboratory notebooks are important records of the work a scientist performs in both academia and industry and are a required component of research at universities and companies. Recording your work and collecting data in a notebook are essential skills in many careers. This documentation is required to prove the authenticity of your work and also allows your work to be repeated by yourself or a colleague. Perhaps more importantly in this course, the act of writing of notebook entry ensures that the student-scientist is prepared prior to the lab experiment, is organized and is actively thinking about what is being done in the laboratory. For example, the *Pre-Lab* section of your notebook will help you structure the details you learned from the required reading and research prior to the experiment and give you an opportunity to plan your procedure. The *Pre-Lab* is also an excellent section to formulate questions and hypotheses that you expect to be able to address during the experiment. The *In-Lab* and *Post-Lab* sections will allow you to demonstrate how well you are able to make observations about your experiment and how well you are able to use those observations and collected data to reach valid conclusions. Finally, we may be conducting original research this semester that may contribute to the publication of a scientific paper. In these cases, accurate lab notebooks are vital in order to record procedures, disseminate data and verify results from many students.

Requirements

For this course, notebooks must be spirally or spine-bound (i.e., no loose-leaf pages) contain a carbon (or carbonless) copy for each page and be consecutively numbered. Acceptable notebooks are available for purchase at the Oakton bookstore. Entries must be completed in ink only; mistakes should be crossed out with a single line. The notebook entries will be graded according to the rubric on the following pages. Only the original pages should be turned in for grading. Keep the carbonless copy for your records. Page numbers must be consecutive for each entry as well as week-to-week for credit. Be sure to use every page; do not leave blank pages in between experiments. No pages that have been inserted or stapled inside the notebook will be graded. The only exception is data that cannot be easily transcribed to the notebook. For example, infrared spectra, gas-chromatographs and Excel graphs may be neatly trimmed and stapled onto the pages of the notebook or attached at the end of the entry. This data should be clearly labeled and titled. Notebooks that have been kept or assembled in three-ring binders will not receive any credit.

Each portion of the lab notebook entry will be completed at different times. The *Pre-lab* section is due the day of the experiment and prior to the beginning of the lab. The *In-lab* is turned in immediately after completing the experiment. The *Post-lab* section is due the following class period, regardless of whether an experiment is scheduled. Recording your procedure and observations will help you think about the experiment and will be the most accurate and detailed when recorded immediately. The *Post-Lab* section may be completed after the lab period; however, it is recommended that you complete this section as soon as possible while the details of the experiment are fresh in your mind. If the lab ends early, this is a great time to complete this section.

II. Anatomy of a Lab Notebook Entry

Front Cover and Table of Contents

Record your name, contact information and course information on the inside cover of your lab notebook. The first page may already be provided for this; however, it's a good idea to replicate this on the inside front cover in case the first page is accidentally torn out. You should also reserve the first couple pages of your lab notebook for a table of contents if those pages are not already provided or if you think you may need more.

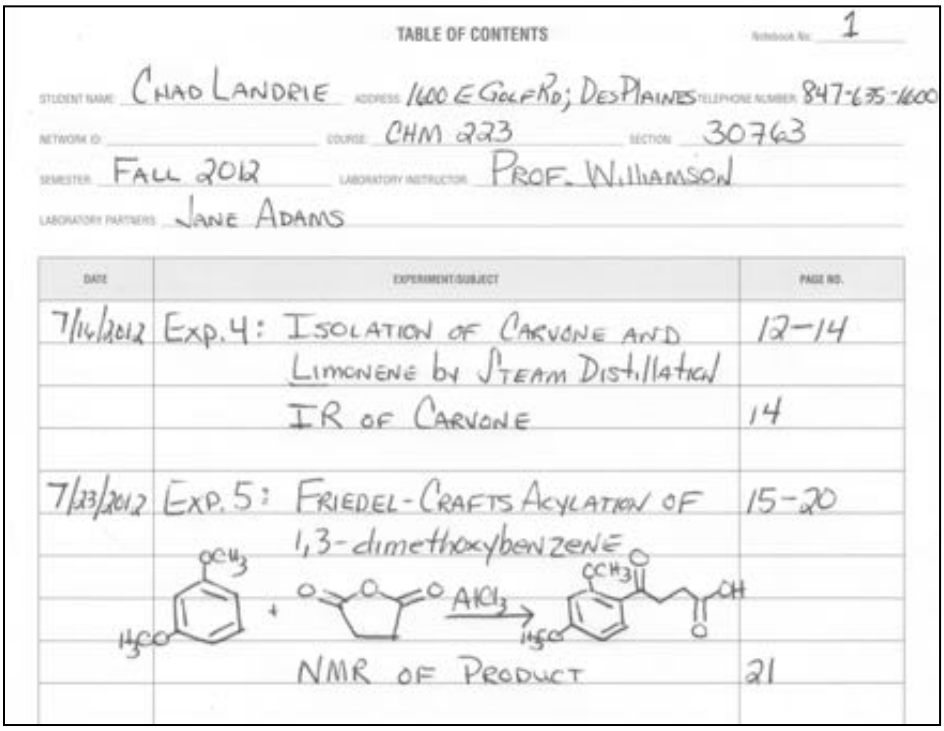


TABLE OF CONTENTS Notebook No. 1

STUDENT NAME Chad Landrie ADDRESS 1600 E Golf Rd; Des Plaines TELEPHONE NUMBER 847-635-1600

NETWORK ID _____ COURSE CHM 223 SECTION 30763

SEMESTER Fall 2012 LABORATORY INSTRUCTOR Prof. Williamson

LABORATORY PARTNERS Jane Adams

DATE	EXPERIMENT/SUBJECT	PAGE NO.
7/16/2012	Exp. 4: Isolation of Carvone and Limonene by Steam Distillation	12-14
	IR of Carvone	14
7/23/2012	Exp. 5: Friedel-Crafts Acylation of 1,3-dimethoxybenzene	15-20
	NMR of Product	21

The *Table of Contents* should contain the date of each entry, the corresponding experiment number in the lab manual, a title and the page numbers in the notebook. The title of your experiment should be descriptive. For synthesis labs, include a reaction scheme below the title and above the chemicals table that summarizes the reaction (see Exp. 5 above). Additionally, data such as infrared spectra, gas-chromatographs and Excel graphs should be referenced within each experiment's entry.

Page Header

The header of each page should contain the experiment number, title (may be abbreviated or shortened), date, name, lab partner name and course (see example below). If page numbers are not already included in your notebook, these should also be added to the top right corner and numbered consecutively on each page. The header must be completed for every page of each entry.

Pre-Lab

The *Pre-Lab* must be completed before the beginning of the experiment and must be turned in before you begin. Each *Pre-Lab* entry may vary slightly, depending on the nature of the experiment. For example, some experiments do not involve chemical reactions and therefore will not have a reaction diagram as in the example below. The required components of a *Pre-Lab* are:

a. Title — The full title of the experiment should be clearly written on the first page of the lab notebook entry. This title may contain more detail than what is written in the header section.

b. Introduction and Purpose: — A brief (5-10 sentences) introduction to the experiment should be written at the top of the page. First, the purpose should state the *chemical* goals and objectives of the laboratory and describe what data will be collected and how that data will be used to arrive at conclusions at the completion of the laboratory. If hypotheses can be made about the outcome of the experiment beforehand, they should be stated here. While the experiments certainly have pedogogical objectives, these should not be included in the purpose. Focus only on the *chemical* investigation. For example:

Correct: To synthesize aspirin by nucleophilic acyl substitution of acetic ahydride with salicylic acid.

Incorrect: To learn correct synthetic procedures and safe handling of hazardous compounds.

Correct: To isolate carvone through steam distillation of caraway seeds; to separate carvone from the distillate through extraction with methylene chloride.

Incorrect: To learn how to setup a steam distllation apparatus; to learn how to perform an extraction.

Second, any important chemical background necessary for interpreting the data or understanding the experiment should be briefly discussed.

c. Chemicals Table or Reaction Table: Make a table of the chemicals (starting materials, reagents, solvents and products) that will be used or produced during the experiment. List the chemicals and their formula, structure, molar mass and physical state. If it will be useful to know a reagent's melting point, boiling point, density or other physical property, include that information as well. For experiments involving chemical reactions or where stoichiometry is important, the quantity of each reagent used should be listed and the number of moles (or millimoles) should be calculated for each quantity. Additionally, for experiments involving chemical reactions, the number of equivalents for each reactant and reagent should be calculated. An equivalent, in this sense, is the molar ratio of that substance to the reactant that is the limiting reagent. In the example below, 1,3-dimethoxybenzene is the limiting reagent. Therefore, the number of millimoles for all reactants and reagents are divided by the number of millimoles of 1,3-dimethoxybenzene used. Equivalents are not calculated for solvents since they do not participate in the reaction. Rather, the concentration of the reactant that is the limiting reagent is calculated for each solvent by dividing the millimoles of limiting reagent by the volume of that solvent.

d. Hazard and Safety Information: In the last column of the chemicals table (above), summarize relevant hazard and safety information. These details can be found by navigating your web browser to http://www.chadlandrie.com/OCCcheminventory. Within the chemical record, read the section titled *Hazard and Safety Information*. Summarize the most important information in 5-10 words.

e. Calculations and Equations: Write out all relevant mathematical equations (include variable definitions) and chemical equations that are applicable to the experiment. For synthesis experiments, the chemical reaction should be written above the *Chemicals Table* for easy reference. Also, write out calculations that can be done ahead of time (e.g., determining the theoretical yield; determining limiting reagent).

f. Anticipated Procedure: Make a numbered list of the tasks that you will complete during the experiment. This list does not have to be exhaustive, but should accurately summarize all aspects of the procedure you are going to complete that day. For example, it is sufficient to state: "Extract aqueous layer twice with methylene chloride." You do not need to state precisely how an extraction is performed. Some of those details will be included in your *Procedure* of the *In-Lab* section. During the lab, you may deviate from your anticipated procedure as the requirements change in response to observations that you are making and data that your are collecting.

g. Pre-Lab Questions: Pre-lab questions for most experiments can be found in the laboratory manual. Your instructor may also ask additional questions during the previous lecture that should be answered in this section.

IN-LAB

The *In*-Lab section contains the details collected during the experiment. It must be turned in immediately after completing the experiment. The *In-Lab* section should be divided into two columns on each page for *Procedure* and *Observations*. The left-hand column will list your actual procedure that you followed during the experiment. The

right-hand column will describe the observations that were made and will also contain data that you collected. The details in the *Observation* column should be written as closely to the procedural step where they were collected as possible. When large amounts of data are to be collected or where it is necessary to construct graphs, a separate page for the *Observations* can also be used. Be sure to label graphs accurately including a title and both the y- and x-axis.

a. Procedure: The procedure listed in this column should be significantly more detailed than the outlined procedure listed in your *Pre-Lab*. Invariably some labs will require steps to be repeated, modified or eliminated based upon observations and collected data. These details should be included. For example, perhaps an unexpected emulsion formed when extracting your aqueous layer (see *Pre-Lab* above) with methylene chloride. You would want to note this in your *Observations* column and then include in your *Procedure* that you added saturated sodium chloride (brine) to break the emulsion.

b. Observations and Data: Here, the more details you record, the more complete this sections will be. Use all of your senses—except taste!—when making observations. Make notes about the state of your reagents (e.g., physical state, color, smell) where appropriate, what happens when reagents are mixed (e.g., color changes, gas evolution) and temperature changes. Also, use this section to accurately record data that the manual asks you to collect during the experiment (e.g., masses, volumes, pH, TLC charts). Use tables where appropriate. Be as precise in your measurements as possible. If calculations are required in order to continue with the experiment, you may also do that math in this section while listing that procedural step in the *Procedure* section. You may also use full pages on subsequent pages if large graphs or tables need to be constructed. To prevent the *Observations and Data* column from becoming cluttered, large data such as infrared spectra, gas-chromatographs and Excel graphs should be attached at the end of this section.

POST-LAB

The primary function of the post-lab section is to analyze the data that was collected during the experiment to reach valid conclusions about the chemical phenomena that were investigated in the experiment. A complete post-lab should contain the following elements:

a. Conclusions: Your *Post-Lab* should contain a medium length paragraph (10-15 sentences) stating the conclusions that you were able to reach during the laboratory experiment. These conclusions should be well supported by the data that you collected and by the calculations that were written in both the *Observations* column and the calculation performed in the *Pre-Lab* ssection above. In other words analyze the data; explain how the results of the experiment(s) led you to the stated conclusions. Also, discuss whether any hypotheses that were postulated in the *Pre-Lab* section were supported or unsupported. Like the *Purpose*, do not state pedagogical conclusions; focus on the chemical conclusions that can be drawn. For example:

Correct: The infrared (IR) spectrum of our product exhibited a broad band at 3200 cm-1, which is characteristic of an alcohol. Additionally, the ketone band at 1620 cm-1 that was present in the starting material was absent in the IR of our product. These observations suggest that benzophenone successfully underwent Gringard addition by phenyl magnesium bromide to provide the tertiary alcohol, triphenylmethanol.

Incorrect: My reaction worked. I learned how to perform a Gringard addition under an inert atmosphere. I also learned how to use IR to determine if an alcohol was formed from a ketone. This reaction taught me a lot about chemical reactions.

b. Calculations: If calculations are required using the data that was collected during the experiment, these should be clearly written here. Where applicable, write the full equation being used at the start of each calculation. Show all work for full credit. Write any new chemical equations that are relevant.

c. Questions: For most laboratory experiments, there will be assigned questions in your laboratory manual. Your responses to these questions is a required portion of your laboratory entry. Your instructor may also ask additional questions during the lab that need to be answered here.

III. Example Lab Notebook Entry

The example below is basic and is intended primarily to illustrate the format and layout. Your entry will likely contain many more details as well as a significant discussion in the conclusion of the *Post-Lab.*

EXP. NUMBER 5 | EXPERIMENT SUBJECT: Friedel-Crafts Acylation | DATE: 7/23/2012 | 01

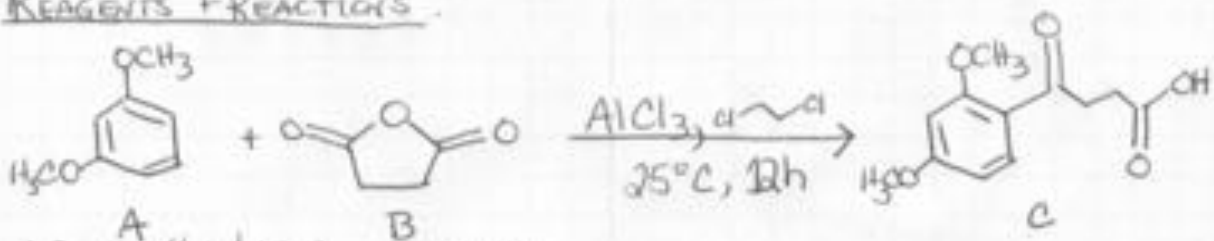
NAME: Chad Landrie | LAB PARTNER: Jane Adams | LOCKER/DESK NO.: 12 B | COURSE & SECTION NO.: CHM 223

TITLE: Friedel Crafts Acylation of 1,3-dimethoxy benzene with succinic Anhydride

PRE-LAB

Introduction: 1,3-dimethoxybenzene is acylated with succinic anhydride. $AlCl_3$ is the Lewis acid used to generate the acylium ion. Because $AlCl_3$ bond irreversibly to succinic Anhydride, it is used stoichiometrically. We will investigate the relationship between the rate of the reaction and the Amount of $AlCl_3$ used.

REAGENTS + REACTIONS



A: 1,3-dimethoxybenzene

B: succinic anhydride

C

Compound Name	Molar Mass (g/mol)	d (g/mL) or C (mol/L)	Reaction Mass/Vol	mmol	Equivalents or Concentration	Hazards
A	138.17	d 1.067	5.0 mL	37.8	1.0	Irritant, toxic to CNS
B	100.07		4.54 g	45.4	1.2	Irritant
$AlCl_3$	133.34		6.63 g	49.2	1.3	Corrosive solid, Irritant
1,2-dichloroethane	98.96	d 1.235	378 mL		37.8 mmol / 378 mL = 0.1 M	Flammable, severe irritant(eyes), carcinogenic
C	178.18					

SIGNATURE: Chad | DATE: 7/23/2012 | WITNESS/TA | DATE

NOTE: INSERT DIVIDER UNDER COPY SHEET BEFORE WRITING

EXP. NUMBER 5 | EXPERIMENT/SUBJECT Friedel-Crafts Acylation | DATE 7/23/2012 | 02

NAME Chad Landrie | LAB PARTNER Jane Adams | LOCKER/DESK NO. 12B | COURSE & SECTION NO. Chm 223

Anticipated Procedure – Short outline

1. Dissolve $AlCl_3$ in 1,2-dichloroethane (Wear gloves!)
2. Cool to 0°C
3. Add succinic anhydride all at once
5. Add 1,3-dimethoxybenzene slowly with a syringe
6. Warm to room temperature.
7. Stir for 12 h
8. Pour into ice
9. Extract with diethyl ether (highly flammable!)
10. Distill away diethyl ether
11. Obtain IR of product

Pre-Lab Questions

1. $AlCl_3$ is a Lewis-acid
2. Diethyl ether and water are immiscible.

In-Lab

Procedure – Actual	Observations
1. Glassware (500 mL rb + syringe) dried in oven	→ • to remove trace H_2O • H_2O reacts w/ $AlCl_3$
2. 6.63g $AlCl_3$ added to rb, then 200 mL 1,2-dichloroethane	→ suspension difficult to stir w/ small stir bar
3. Cooled stirring mixture to 0°C w/ ice bath	
4. Add 3.9g succinic anhydride	→ • solution color turned deep red • color of acylium ion?
5. Stir 5 min	

SIGNATURE Chad | DATE 7/23/2012 | WITNESS/A | DATE

EXP. NUMBER 5 | EXPERIMENT/SUBJECT Friedel Crafts Acylation | DATE 7/23/2012 | 03

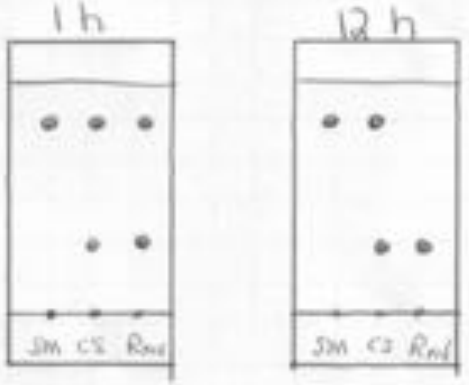
NAME Chad Landrie | LAB PARTNER Jane Adams | LOCKER/STN NO. 12D | COURSE & SECTION NO. CHM 223

Procedure Continued

6. Added 1,3-dimethoxybenzene
7. Took TLC at 1h
8. Took TLC at 12 h
9. Pour rxn into ice
10. Let ice melt
11. Extract water layer w/ 3 x 50 mL Et_2O
12. Dry organic layer w/ Na_2SO_4
13. Filter
14. Remove solvent + IR

Observations Cont

→ red color disappeared



Solvent = 1:1 EtOAc/hexanes
Stain = $KMnO_4$

↳ emulsion upon extraction, try EtOAc next time

Post-Lab

Questions/Conclusions

1. White smoke formed was HCl/H_2O
2. Carboxylic acids are more polar than esters.

Data

Mass C: 5.20 g
% yield: 81%
m.p.: 220-222°C
IR: 2850, 1645, 1602, 3450 (broad) cm^{-1}
(see attached)

Calculations

$$5.00\text{ g A} \times \frac{1\text{ mol A}}{137.17\text{ g}} \times \frac{1\text{ mol C}}{1\text{ mol A}} \times \frac{178.8\text{ g C}}{1\text{ mol C}} = 6.45\text{ g C (theoretical yield)}$$

$$\%\text{ Yield} = \frac{\text{Actual}}{\text{theoretical}} \times 100 = \frac{5.20}{6.45} \times 100 = 81\%$$

SIGNATURE	DATE	WITNESS/TA	DATE

IV. Laboratory Notebook Grading Rubric

STUDENT NAME:	**COURSE/SECTION:**
EXP:	**SHORT TITLE:**

	Excellent	Good	Fair	Poor	Pts
Organization	**2 pts:** The notebook entry is clearly divided into the *Pre-Lab*, *In-Lab*, and *Post-Lab* sections with their respective subsections. A title is present on the first page of the entry. Every page of the entry contains a completed header and is numbered consecutively. A grading rubric is stapled to the front of the Pre-Lab.	**1 pt:** One or two subsections of a major division are missing, out of order or not labeled clearly. May contain an incomplete header: missing a title, non-consecutive page numbers, name and date on each page or student signature on every page. May contain black pages. Grading rubric may be missing.		**0 pts:** More than two key elements of organization are missing or not clearly identified.	
Pre-Lab	**2 pts:** The *pre-lab* is well written, organized and neat. It contains all required elements: title, introduction, chemicals table, equations/reactions and anticipated procedure. Every element is thorough and correct. The instructors signature is found at the end of the *Pre-Lab*.	**1.5 pts:** All elements are present, but there are minor errors, misinformation or slight omissions in one or two of these elements.	**1 pt:** One or two elements are missing, contain several errors, or significant omission of required detail is noted. Introduction contains little detail.	**0 pts:** More than two elements are missing or have not been completed. Instructor's signature is missing..	
Procedure (In-Lab)	**2 pts:** The procedure is thorough and contains details that were specific to the *actual* experience in the laboratory. Deviations and modifications errors are all recorded in a chronological sequence of events. It is clearly written and legible.	**1 pt:** Generally, the procedure is well written and thorough, but may be missing some key steps that were *actually* performed (or should have been performed) in the laboratory. Some changes and modifications that were required during the laboratory may not be recorded. May be difficult to follow or read.		**0 pts:** The procedure mimics the *Pre-Lab* outline or precisely lists the manual's steps with few notes on *actual* events. May be illegible.	
Observations (In-Lab)	**2 pts:** Observations are plentiful and clearly noted for each experiment with details including color changes, precipitation, temperature, m.p., b.p., mass, etc. Charts and graphs are recorded where necessary. Diagrams (e.g., TLC) are drawn where needed. Data is collected, including spectra, tables and charts.	**1 pt:** Observations are plentiful and clearly noted. Some observational detail may be missing or some data collection/calculations may contain minor errors (e.g., missing titles, incorrect axis lable). All data is recorded clearly and correctly including charts, graphs, tables, spectra, m.p., b.p., mass.		**0 pts:** Required data may be missing. Or, observations are minimal with little depth. Details do not reflect the lab or do not support the procedure.	
Questions, Calculations, and Conclusions (Post-Lab)	**2 pts:** A conclusion is present and accurately analyzes the data collected. Conclusions demonstrate thought and critical thinking about the experiement. Explanations are provided for the results and the data is used to support or invalidate the original hypotheses postulated in the *Pre-Lab*. All calculations are correctly and clearly written out with all work shown. All *Post-lab* questions are correctly answered. The *Post-Lab* is legible and each subsection easily located.	**1.5 pts:** One calculation or question is done incorrectly or contains minor errors. All work is shown for all. One expected conclusion is missing or is not fully supported by the data. Data mostly analyzed correctly.	**1 pt:** More than two calculations and/or questions are done incorrectly. More than one conclusion may be missng, incomplete, or unsupported. Data may be only loosely tied to conclusions or misinterpreted.	**0 pts:** Group work = cheating. Work individually. More than 3 calcs and/or questions are done incorrectly. Paraphrases manual with little data analysis; few to no conclusions or may be wrong. Data is not used or is misinterpreted.	
Comments.				Total Score →	