Appendix C

Laboratory Notebook Maintenance

Maintenance of a scientific laboratory notebook is a significant portion of your grade. The course website (http://www.uncw.edu/chem/online.html) provides a detailed list of entries required for each experiment.

Basic Rules

- A duplicate carbonless notebook is a required text for the course. The notebook can be used for both semesters of the lab course.
- All entries must be made in black or blue ink ONLY. No pencil or colored ink.
- Do not skip pages or tear out the original copies of the notebook.
- All entries should be dated!
- It is important to write things in the notebook as they happen during the experiment, not after the lab period is over!
- Do not scribble through or white-out any mistakes. It is best to cross through the mistake with a single line.

Components of a Laboratory Notebook

The following components should be contained for each experiment, along with any additional material noted on the course website:

- Title and date
- Objectives
- Physical data
- Procedure outline
- Data and observations

For each experiment, there will be a portion to be completed before coming to lab, called the **PRE-LAB**, and a portion to be completed during the experiment, called the **IN-LAB**. The pre lab portion of a laboratory notebook should be entered on a separate page, and the yellow copy of this page will be collected at the beginning of each lab for grading. Students will not be allowed to perform the experiment without having a complete pre lab. The copy of the in lab portion of the lab notebook will be submitted after the experiment is complete.

LAB NOTEBOOK REQUIREMENTS

(Your laboratory notebook should contain the following sections in this order.)

PRE-LAB NOTEBOOK

1. **TITLE AND DATE**
2. **OBJECTIVE:** In a sentence or two, briefly state the purpose of the experiment.
3. **CHEMICAL EQUATION:** For synthesis experiments only.
4. **TABLE OF PHYSICAL DATA:** Include a table of physical data, including the compound name, molecular weight, boiling/melting point, density, and hazards for all *organic* compounds and *hazardous inorganic reagents* used in the experiment. A link to the physical data will be available on the course website, but there are also links to websites such as www.chemexper.com and www.hazards.com, which can be used when physical data are not given. Be sure to credit the source of the physical data with the proper footnote.
5. **REFERENCE TO PROCEDURE**: You should read the experimental procedure thoroughly before coming to lab. However, for the pre lab notebook assignment, you should simply include a reference to the lab manual, including title, author(s), edition, and specific page numbers where the procedure can be found.

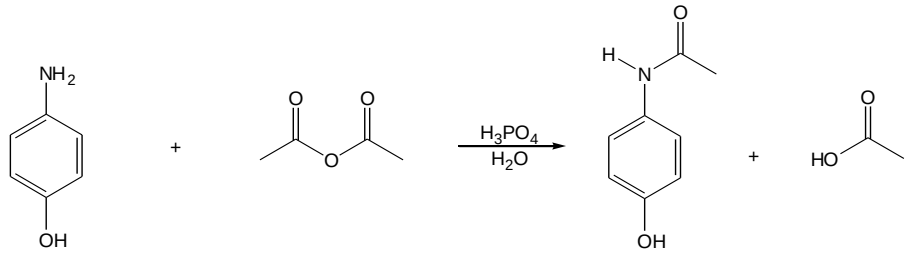
SAMPLE PRE-LAB NOTEBOOK ENTRY

Experiment title and number				Date
Exp. 30 Synthesis of Acetaminophen				*8/08/08*
Name	Course	Section	Lab Partner	
Jane Doe	*211*	*201*	*Jim Doe*	

Objective

The objective of this experiment is to synthesize acetaminophen using p-aminophenol and acetic anhydride, to purify the product by recrystallization, and to determine purity using melting point and HPLC analysis.

Chemical Equation *(can be handwritten!)*



p-aminophenol (reactant A) — *acetic anhydride (reactant B)* — *acetaminophen (desired product)* — *acetic acid (side product)*

Table of Physical Data *(from www.chemexper.com)*

Name	*MW (g/mol)*	*BP (°C)*	*MP (°C)*	*D (g/mL)*	*Hazards*
p-aminophenol	*109.13*	*-*	*188*	*-*	*Harmful, environmental hazard*
Phosphoric acid	*98.00*	*158*	*-*	*1.68*	*Corrosive*
Acetic anhydride	*102.09*	*140*	*-*	*1.09*	*Corrosive*
Ethyl Acetate	*88.11*	*77.5*	*-*	*0.902*	*Flammable, harmful*
acetaminophen	*151.16*	*-*	*168-172*	*-*	*Harmful*

Experimental Procedure *(from CHML 211-212 Laboratory Manual for Organic Chemistry, Fall 2009, p. 7)*

LAB NOTEBOOK REQUIREMENTS

(Your laboratory notebook should contain the following sections in this order.)

IN-LAB NOTEBOOK

6. **EXPERIMENTAL PROCEDURE:** In *paragraph form*, describe the procedure that you ***actually*** followed during the lab. Include any volumes or weights of chemicals used during the experiment. This paragraph should be written in past tense, passive voice.

7. **DATA AND CALCULATIONS:** Note any weights, volumes, experimental melting points, and observations that you make during the experiment. Be sure to include **DATE AND TIME** that information is recorded in this section before writing in data. A list of required entries for each experiment will be provided on the course website.

FINAL LAB REPORT

8. **EXPERIMENTAL RESULTS** (on final lab report): In addition to recording data in your lab notebook as you collect it, the experimental results are generally entered into tables which have been prepared on the final lab report for each experiment. All tables should be completed using black/blue ink ONLY. Pencil and colored ink are unacceptable in the final data tables.

9. **DISCUSSION AND CONCLUSIONS** (on final lab report): The discussion and any conclusions reached during the experiment are presented in the form of questions on the final lab report for each experiment. These questions can be completed using pencil or pen.

SAMPLE IN-LAB NOTEBOOK ENTRY

The following is an example of an experimental procedure for the synthesis of acetaminophen. The laboratory notebook example that follows is based on this experimental procedure. It is to be used as a reference for how to paraphrase a procedural outline for a synthesis experiment in your laboratory notebook. The data and calculations are also shown in the example following the written procedure.

Synthesis

- Fill a 400 mL beaker half full with tap water. Place the beaker and water on a hot plate and bring to a boil.
- Weigh 1.5 g of *p*-aminophenol into a 125 mL Erlenmeyer flask and add 25 mL of deionized water. Swirl to mix.
- Add 20 drops of phosphoric acid, and swirl the flask until all of the solid dissolves. If necessary, add a few more drops of phosphoric acid.
- Remove the beaker of water from the hot plate and, using a clamp, immerse the flask in the beaker of hot water.
- Carefully add 2.0 mL of acetic anhydride to the flask. Leave the flask in the warm water for 10 minutes.
- Remove the flask from the water bath and allow it to cool to room temperature. When cool, place the flask in an ice bath to induce crystallization. Leave the flask in the ice-water bath for 20 minutes. If after 20 minutes no crystals have formed, scratch the inside of the flask with a glass rod.
- Set up a vacuum filtration apparatus. Collect the crude product in a Büchner funnel. Wash the crystals with 10 mL of ice-cold deionized water. Leave under vacuum for 10 minutes.

Purification

- Transfer the crude acetaminophen crystals to a 100 mL beaker. Add 20 mL of deionized water and heat on a hot plate until the crystals dissolve. If the water begins to boil and crystals still remain, add an additional 10 mL of deionized water.
- Remove the beaker from the hot plate and allow it to cool to room temperature. When cool, place the beaker in an ice-water bath for 20 minutes.
- Set up a vacuum filtration apparatus. Preweigh a small filter paper and collect the recrystallized product in a Büchner funnel. Wash the crystals with 2 mL of ice-cold deionized water. Leave under vacuum for 5 minutes.
- After several minutes under vacuum, remove the entire filter cake from the Büchner funnel and transfer to a preweighed larger filter paper.
- After preparing and HPLC sample (see *Analysis* below), submit your sample to your instructor to dry until the next lab period.
- During the next lab period, obtain the final mass of your acetaminophen and calculate the percent yield. Also, perform a melting point analysis (see *Analysis* below).

SAMPLE IN-LAB NOTEBOOK ENTRY

Analysis

HPLC Analysis:

- Prepare an HPLC sample of your recrystallized product by placing two or three crystals in an autoanalyzer vial and dissolving in ethyl acetate (HPLC solvent).
- Submit for analysis. When the sample chromatogram is returned, identify any compounds present in your sample using the provided standard. Also, determine the purity of your sample based on an HPLC analysis.

Melting Point Analysis:

- Prepare two melting point capillaries of your recrystallized acetaminophen.
- Perform a melting point analysis using the Mel-Temp apparatus as described by your instructor.
- Determine the purity of your product by comparing the experimental melting point of your acetaminophen to the literature melting point of the pure substance.

Experiment title and number			Date
Exp. 30 Synthesis of Acetaminophen			*8/08/08*
Name	Course	Section	Lab Partner
Jane Doe	*211*	*201*	*Jim Doe*

Experimental Procedure *(8/08/08, 2:48pm)*

Around 200 mL of water was brought to a boil in a 400 mL beaker using a hotplate. Deionized water (25 mL) and p-aminophenol (1.52 g) were added to a 125 mL flask and swirled to mix. Phosphoric acid (20 drops) was added to the reaction flask, and swirled to mix. Once the solid had dissolved, the water bath was removed from the hotplate, and the reaction flask was immersed in the hot water bath. At this time, acetic anhydride (2.0 mL) was added. The reaction flask remained in the water bath for an additional 10 minutes, at which time it was allowed to cool to room temperature, followed by 20 minutes in an ice water bath until crystals appeared. The resulting white solid was collected using vacuum filtration, and the crystals were rinsed with ice-cold deionized water (~10 mL).

To purify the solid acetaminophen, the crystals were transferred from the filter paper to a 100 mL beaker. Deionized water (20 mL) was added directly to the beaker, and heated on a hot plate until the crystals dissolved. Once dissolved, the beaker was removed from the hotplate and allowed to cool to room temperature at which time the recrystallized solid appeared. After cooling in an ice-water bath for 20 minutes, the purified solid was collected using vacuum filtration, rinsed with ice-cold deionized water (~2 mL), and left under vacuum for 5 minutes.

After purification, the pure solid was transferred to a larger preweighed filter paper for storage. An HPLC sample was prepared by transferring a few crystals to an autoanalyzer vial, dissolving in HPLC solvent (ethyl acetate) and submitted for analysis. Finally, the pure solid was secured and submitted to the instructor to dry until the next lab period. During the next lab period, the pure solid will be weighed, the percent yield will be determined, and melting point analysis will be performed.

Data and Calculations

Weight of p-aminophenol: 1.52 g
Volume of acetic anhydride: 2.0 mL
Volume of phosphoric acid: 20 drops
Weight of small filter: 0.21 g
Weight of large filter: 0.82 g
HPLC vial slot #: 8
Product Appearance: white solid

Theoretical yield calculation:

$$\frac{1.52\text{ g RCT A}}{} \left| \frac{1\text{ mol RCT A}}{109.13\text{ g}} \right| \frac{1\text{ mol PROD}}{1\text{ mole RCT A}} \left| \frac{151.16\text{ g PROD}}{1\text{ mol PROD}} \right. = 2.11\text{ g} \ldots \text{(limiting reagent)}$$

$$\frac{2.0\text{ mL RCT B}}{} \left| \frac{1.09\text{ g}}{1\text{ mL}} \right| \frac{1\text{ mol RCT B}}{102.09\text{ g}} \left| \frac{1\text{ mol PROD}}{1\text{ mol RCT B}} \right| \frac{151.16\text{ g PROD}}{1\text{ mol PROD}} = 3.23\text{ g}$$