

Writing an Organic Synthesis Lab Report

Components of a Laboratory Notebook

The following components should be contained for each experiment, along with any additional material required by your instructor.

- Title and date
- Introduction (purpose, reaction)
- Physical data (including calculations)
- Procedure outline
- Data and observations
- Discussion of results (conclusions)

Prelab

Title and Date

Give the title of the experiment and the date on which it is done.

Introduction

In a sentence or two, state the purpose of the experiment. If the experiment is a preparative experiment, the introduction also includes the balanced equation for the reaction.

In organic chem, there are different types of experiments: technique and preparative. A technique experiment is one in which you are performing a technique for the first time and studying its details, for example, distillation and extraction. A preparative experiment is one in which a compound is synthesized from other reagents.

Physical Data

List the molecular weight, melting point, boiling point, density, solubility, and hazards of all pertinent chemicals used in the experiment. You can find this information in the CRC Handbook of Chemistry and Physics. (We have this book at school, and a CD-ROM version as well.) Or, you can find the information on the Internet (see: [Hazard and Physical Data for Compounds](#) page). **Chemfinder.com** is an excellent site to find this data. The physical data are most conveniently presented in tabular form, although in a preparative experiment you may put the amounts of reactants and products under the balanced equations for the reaction.

Calculate the amounts of reactants (or compounds to be purified) in moles and grams or mL (as applicable). In a preparative experiment, calculate the limiting reagent and the theoretical yield of the product. Be sure to include your calculations for these values.

Procedure

Briefly summarize the procedure to be followed, preferably either as an outline or as a flow chart. You do not need to write out the procedure in complete sentences and do not copy directly from the Lab Manual. All you need is a brief but complete listing of what you plan to do in the lab. The first time you do a technique, such as distillation, include in the procedure section a description of how to assemble the apparatus and how to conduct the distillation. In later experiments, it will be sufficient to state only that the liquid was distilled.

Data and Observations

Your observations of the experiment as it progresses is important, new information. Write these observations (color changes, appearance of crystals, formation of an emulsion, boiling temperatures, test results, etc.) in your notebook as you do the experiment. Also record the weights of reagents and products and tare weights in this section.

In general, you do not need to re-write the Procedure section in these observations, instead, you may state that “the procedure was carried out as planned” or “the procedure was carried out as planned except” At times, however, you may have to write the procedure out partially. For instance, if you state “the solution turned green,” you will have to write out enough of the procedure so that your instructor will know at what step in the reaction the solution turned green. As a guideline, consider that from the procedure and data and observations sections, any chemist should be able to duplicate your experiment. With this in mind, be thorough but include only pertinent information.

Discussion of Results

This is the section in which you interpret the data obtained in the previous section. For instance, indicate the amount of purified compound that you obtained and how the purity and identity of the compound was assessed. In a preparative experiment, state the percent yield. Include and discuss instrument printouts, such as GC traces and IR spectra (if you have them). In this section, you can state whether or not the procedure was a good method for making the desired compound; if not, try to make suggestions to improve the method for future experimenters. Be sure to include a discussion of possible sources of error, and how that error would affect the overall yield.

Sample Lab Report

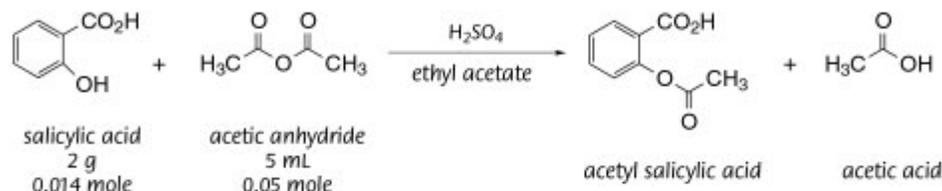
The next 4 pages show a sample preparative organic chemistry lab report. You do not need to include the header and footer on each page. However, you should include the information in the header on the first page.

Adapted from <http://orgchem.colorado.edu/hndbksupport/labnb/labnb.html>

EXP. NUMBER 1	EXPERIMENT/SUBJECT Organic Chem 2 Lab	DATE 2/29/00	1
NAME John Doe (TA: Gregg N. Yard)		LOCKER NO. 55	COURSE & SECTION NO. chem 3341-111

The Preparation of Aspirin

The purpose of this experiment is to synthesize aspirin (acetyl salicylic acid) from salicylic acid and acetic anhydride.



The limiting reagent is salicylic acid. The theoretical yield of acetyl salicylic acid is 2.52 g.

Physical Data:*

	MW	mp	bp	density	solubility	hazards
salicylic acid	138	157-9°	—	—	al, eth, ace	toxic
acetyl salicylic acid	180	135-6°	—	—	al, eth, chl	irritant
acetic anhydride	102	—	138°	1.08	—	corrosive, lachrymator
acetic acid	60	—	117-8°	1.049	—	corrosive
sulfuric acid	98	—	—	1.84	—	corrosive
ethyl acetate	88	—	77°	0.90	—	flammable

*Data from the CRC, 70th ed.

Calculations:

2 g salicylic acid (1 mole/138 g) = 0.014 moles

5 mL acetic anhydride (1.08 g/mL) = 5.4 g then,

5.4 g (1 mole/102 g) = 0.05 moles

thus salicylic acid is present in the lesser molar amount and is the limiting reagent

therefore the theoretical yield of acetyl salicylic acid is 0.014 moles, or

0.014 moles (180 g/mole) = 2.52 g

Procedure

From: Experiments for Organic Chemistry, Chem 3341, Spring 2000, pp. 20-25

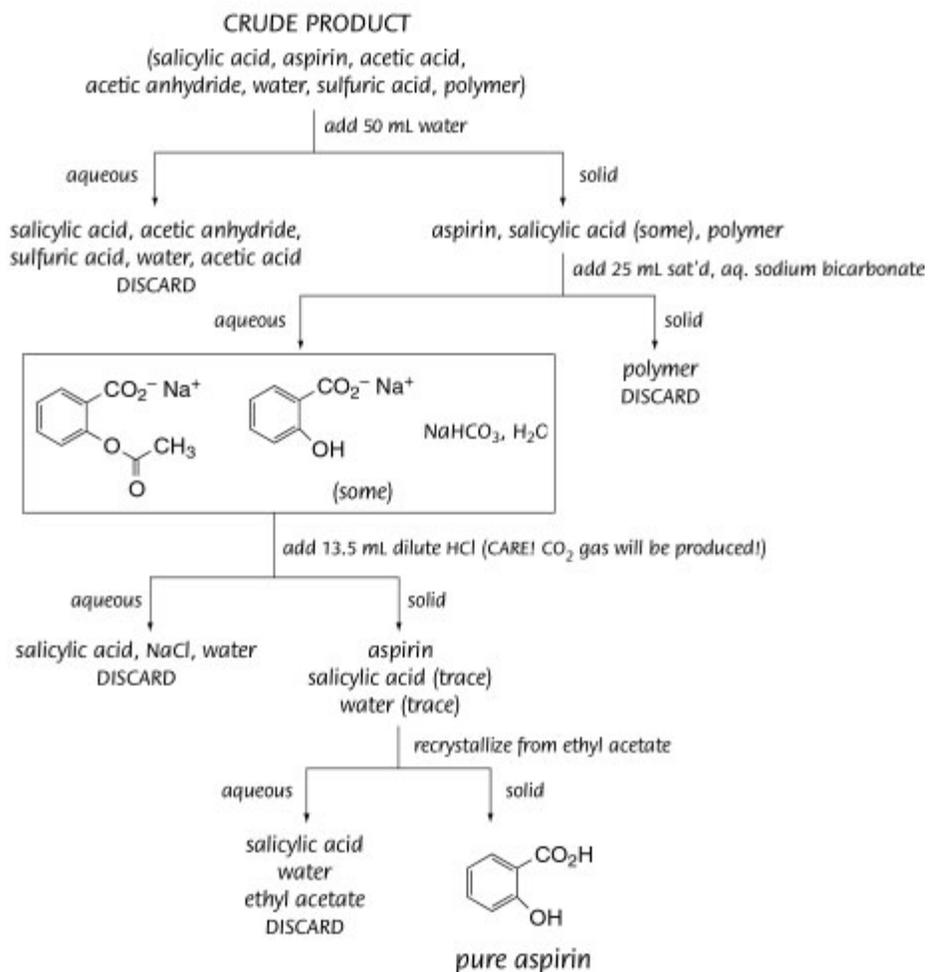
- 1) Mix salicylic acid and acetic anhydride in a 125 mL Erlenmeyer flask, add 5 drops H₂SO₄.
- 2) Heat on steam bath for 10 min, then cool.
- 3) Add 50 mL water and cool on ice.
- 4) Collect product by vacuum filtration.
- 5) Air dry the crude product crystals and determine a crude yield.
- 6) Purify as in the flow chart ~~below~~ on the next page.

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NAME John Doe (TA: Gregg N. Yard)		LOCKER NO. 55	COURSE & SECTION NO. chem 3341-111

The Preparation of Aspirin (con't)

6) Continued: flow chart for purification of crude product.



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The Preparation of Aspirin (con't)

Data and Observations

wt of salicylic acid + paper: ~~2.30 g~~ 2.43 g
 wt of paper: 0.43 g
 salicylic acid: 2.00 g

- 1) On mixing, it took a few minutes for everything to go into solution. The addition of sulfuric acid caused some fizzing.
- 2) Heated for about 15 min instead of the planned 10 min.
- 3) After adding the water and cooling, no crystals appeared. On the suggestion of my TA, I scratched the flask with a glass rod, chilled it on ice for 10 more min, and finally a lot of slightly tan crystals appeared.
- 4) Quite a lot of solid, lightly tan, was collected on the filter paper
- 5) Let dry for 10 min.

crude product + watchglass: 32.02 g
 watchglass: 30.10 g
 crude product: 1.92 g
 % yield of crude product: $1.92 \text{ g} / 2.52 \text{ g} = 76\%$
 mp = 125-129°

- 6) When I followed the scheme in the flow chart for purification of the crude product, I noticed that when I added the sodium bicarbonate, the product turned yellow. I suspected a contaminated (dirty) beaker. So, I treated the mixture with Norite pellets. A clear solution resulted. The purification scheme was followed without mishap through the recrystallization from ethyl acetate.

crude product + watchglass: 31.80 g
 watchglass: 30.10 g
 crude product: 1.70 g
 % yield of crude product: $1.70 \text{ g} / 2.52 \text{ g} = 67\%$
 mp = 133-135°

Conclusion

The yield of purified aspirin was 1.70 g or 67% yield. Although an acceptable value, future experimenters could take steps to better the yield, perhaps by running the reaction for longer than 15 min to encourage more product formation, or by more carefully rinsing the flask when transferring crystals. Also, some product may have been (con't)

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EXP. NUMBER 1	EXPERIMENT/SUBJECT Organic Chem 2 Lab	DATE 2/29/00	4
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The Preparation of Aspirin (con't)

Conclusion (con't)

lost by the Norite step (added to remove the colored contaminant). I'd suggest carefully checking the cleanliness of all glassware before beginning the purification step to eliminate the need for this step and thus to improve yield.

The wide range and low value of the mp of the crude product indicates that before recrystallization, the aspirin was not very pure. After recrystallization, the small mp range of aspirin (133-135°) indicates a pure compound. This value correlates well with the literature value (135-136°) for the mp of aspirin. From this data, it is likely that the compound isolated is aspirin, although further tests such as mixed melting points and spectroscopic data would be required to prove that it is aspirin.

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