

Experiment 8: Synthesis of acetylsalicylic acid

Work individually.

• Pre-lab:

Read: Experiment 8 (pp. 63 – 66), Technique 2, Figure 2.2 (p. 563)

Prepare for class on Thursday, February 21:

Write a one-sentence purpose. Don't forget the word "characterize".

Under the "Purpose" section, write the overall chemical equation for this reaction.

Under the "Materials and methods" section, organize the chemical information into a table (see figure 2.2, p. 563) that lists physical constants of the reactants and products. You will have to look up values in the Merck Index or CRC or on-line. Add three more columns to the table titled "Intended (or expected) amount/amount used (or obtained)", "Intended (or expected) moles/moles used (or obtained)" and "Health and safety issues".

Calculate the moles of the reactants and products, and enter that into the table. Use the amounts given in the procedure (p. 65) to fill in the reactants intended amounts. Do a calculation to figure out what the expected yield of the product will be.

Include the expected yield calculation here.

Cite the source of your physical constant information.

Separation scheme: Simply state which purification technique you've learned so far that you are going to use to isolate the product of this experiment.

For the equipment part, include a sketch of the setup.

"Procedure" section: You may copy (or photocopy) the procedure in PLKE (pp. 65-66) into your lab notebook.

"Data" section: **Prior to lab**, make a data table to record actual amounts (the clever will figure out a way to append this to the table under "Materials and methods") of reactants and products, as well as melting points.

• During lab:

After weighing the dry yield, perform the melting point and ferric chloride tests as indicated in PLKE, p. 66. Record your observations.

Obtain NMR spectra of the salicylic acid and your acetylsalicylic acid.

• **Post-lab:**

In the “Results” section, include copies of both NMR spectra, along with peak assignments.

Calculate the percent yield of acetylsalicylic acid.

In the “Conclusions” section:

- Comment about the percent yield, and give sources of significant yield loss.
- Comment about the purity of your product, using the melting point, ferric chloride test and NMR results. Make a special note to address whether there was any unreacted starting material in your final product.

• **Lab Result Report: (Due Thursday, February 28 at the beginning of lab)**

Photocopy the lab, all parts, including the NMR spectra.

No abstract is needed.

Answer questions:

End of Experiment 8 (p. 66): 1, 2, 7