

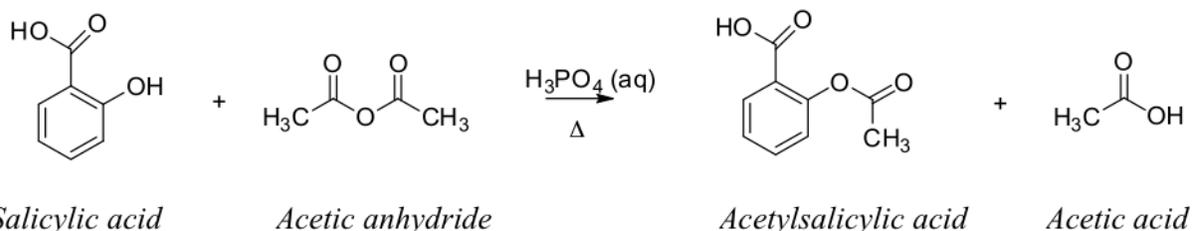
## Experiment

# 16

## The Synthesis and Analysis of Aspirin

Acetylsalicylic acid is the chemical name for aspirin, the ubiquitous pain reliever. One of the compounds used in the synthesis of aspirin is salicylic acid, which is itself a pain reliever that was known to many ancient cultures, including the Native Americans who extracted it from willow tree bark. Salicylic acid is extremely bitter tasting, and frequent use can cause severe stomach irritation. The search for a milder form of this pain reliever led to the successful synthesis of acetylsalicylic acid by the German chemist Felix Hoffmann in 1893.

Your two primary objectives in this experiment will be to synthesize and analyze aspirin. There is more than one way to synthesize aspirin; in this experiment, you will react acetic anhydride with salicylic acid in the presence of phosphoric acid (which acts as a catalyst). The reaction equation is shown below.



You will conduct two tests of your synthesis to verify that you did indeed make aspirin, and to determine its relative purity. First, you will measure the melting temperature of a sample of your product, using a Melt Station. Second, you will use a Spectrophotometer to test the absorbance of salicylic acid impurity in your aspirin sample after it has been complexed with  $\text{Fe}^{3+}$  from iron (III) nitrate solution.

### OBJECTIVES

In this experiment, you will

- Synthesize a sample of acetylsalicylic acid (aspirin).
- Calculate the percent yield of your synthesis.
- Measure the melting temperature of your aspirin sample.
- Conduct a spectrophotometric analysis of your aspirin sample.

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# MATERIALS

### Part I Synthesis Materials

50 mL Erlenmeyer flask	solid salicylic acid
two 10 mL graduated cylinders	85% phosphoric acid solution, H <sub>3</sub> PO <sub>4</sub>
25 mL graduated cylinder	liquid acetic anhydride
250 mL beaker	distilled water
vacuum filtration apparatus	cold distilled water
spoon, spatula, or rubber policeman	balance
ice bath	watch glass
hot plate	Wide-Range Temperature Probe or
plastic Beral pipet or eyedropper	thermometer

### Part II Melting Temperature

LabQuest or computer interface	aspirin crystals (from Part I)
LabQuest App or Logger <i>Pro</i>	tissues (preferably lint-free)
Vernier Melt Station	mortar and pestle (optional)
glass capillary tubes, one end closed	

## PROCEDURE

### Part I Synthesize Aspirin

1. Obtain and wear goggles. Protect your arms and hands by wearing a long-sleeve lab coat and gloves. Conduct this reaction in a fume hood.
2. Measure out 2.0 g of salicylic acid into a 50 mL Erlenmeyer flask.
3. Add 5.0 mL of acetic anhydride and 5 drops of 85% phosphoric acid solution. Swirl the mixture. If necessary, use a sparingly small amount of distilled water to rinse down any bits of solid that may be on the inner walls of the flask. **CAUTION:** *Handle the phosphoric acid and acetic anhydride with care. Both substances can cause painful burns if they come in contact with the skin.*
4. You are now ready to begin the synthesis of aspirin.
  - a. Prepare a 70–80°C hot-water bath using a 250 mL beaker on a hot plate. Monitor the water temperature using a Temperature Probe or thermometer.
  - b. Hold and partially submerge the 50 mL flask and contents in the water bath.
  - c. Heat the mixture in the hot-water bath for 15 minutes, or until the mixture ceases releasing vapors. Stir the mixture occasionally during heating. Add 2 mL of distilled water about 10 minutes into the heating.
5. Crystallize the aspirin.
  - a. When you are confident that the reaction has reached completion (no vapors appearing), carefully remove the flask from the hot plate and add 20 mL of distilled water.
  - b. Allow the mixture to cool to near room temperature. Transfer the flask to an ice bath for about five minutes. As the mixture cools, crystals of aspirin should form in the flask.

6. Wash the synthesized aspirin.
  - a. Set up a vacuum filtration. Be sure to weigh and record the mass of the filter paper to the nearest 0.001 g before filtering the solid.
  - b. Filter the mixture with vacuum suction.
  - c. When most of the liquid has been drawn through the funnel, turn off the suction and wash the crystals with 5 mL of cold, distilled water.
  - d. After about 15 seconds, turn the suction back on. Wash the crystals with cold, distilled water twice more in this manner.
  - e. Gently transfer the filter paper with your product onto the watch glass to air dry. As directed by your instructor, either direct a gentle stream of air (low flow) to help dry the solid, or let them air dry until the next lab period.
  - f. Weigh the dried recrystallized product on the filter paper and record the mass to 0.001 g.
7. Determine the mass of your dry aspirin sample and record in the data table.

**Part II Test the Melting Temperature of an Aspirin Sample**

8. Obtain a small amount of your synthesized aspirin from Part I. The solid should be in a powdered form. If it is not, use a mortar and pestle to carefully grind the solid to a powder. Pack the capillary tube.
9. Check the control dial on the Melt Station to confirm that it is in the Off position. Connect the Melt Station power supply to a powered electrical outlet.
10. Connect the Melt Station to a LabQuest or to a computer interface. Choose New from the File menu of the data-collection program
11. Carefully insert the capillary tube of solid into one of the sample holders of the Melt Station.
12. Begin collecting melting temperature data. In the first trial, you will want to observe the melting process and make a *rough estimate* of the melting temperature of your unknown sample.
13. When you have determined the approximate melting temperature range for the sample, stop data collection and turn the dial to the Fan/Cooling setting. Record the melting temperature range in your data table.
14. Now that you have a rough idea of the melting temperature, a more accurate determination of the melting temperature can be made. Prepare a sample in a capillary tube and determine the melting temperature of the sample:
15. When finished, stop data collection and turn the dial to the Fan/Cooling setting. Record the melting temperature range in your data table.
16. At the end of the experiment turn the control dial on the Melt Station to Off. Dispose of the capillary tubes as directed by your instructor.
17. Complete the Data Analysis section before exiting Logger *Pro* or the LabQuest App. Print a copy of your graph and/or save your data, as directed by your instructor.

## Experiment 16

NAME: \_\_\_\_\_

### DATA TABLE

#### Part I Synthesis of Aspirin

Mass of salicylic acid used (g)	
Volume of acetic anhydride used (mL)	
Mass of acetic anhydride (1.08 g/mL) used (g)	
Mass of aspirin and filter paper (g)	
Mass of filter paper (g)	
Mass of aspirin synthesized (g)	

#### Part II Data Analysis

	Synthesized aspirin	Commercial aspirin
Melting temperature range (°C)		
Mean melting point (°C)		

Actual (known) melting point of pure acetylsalicylic acid from online source (°C)	
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1. Calculate the percent difference in melting points,  $\frac{| \text{known} - \text{experimental} |}{\text{known}} \times 100$

i. Commercial aspirin

ii. Your synthesized aspirin

2. Calculate the theoretical and percent yield of your synthesized aspirin.