Experiment 6. Preparation of Aspirin and Analysis

1st week

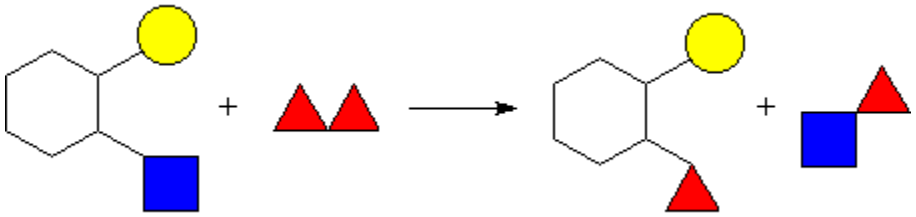
Experimental Procedure 402

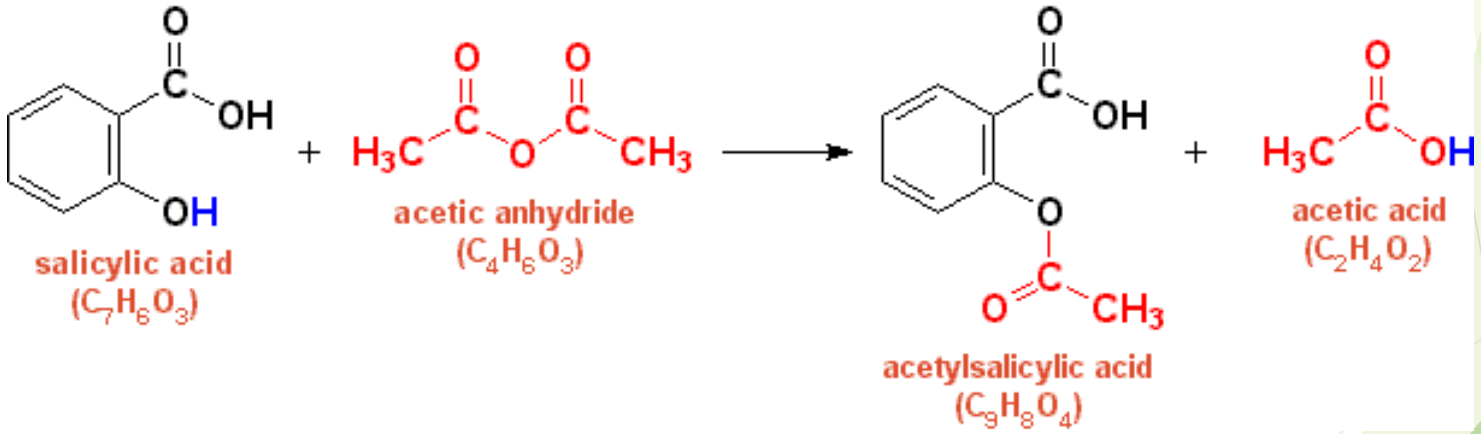
Overview

Crystalline aspirin is synthesized and then purified by the procedure of recrystallization. The melting point and the percent purity of the aspirin are determined, the latter by titration with a standardized NaOH solution.

PART A. Preparation of Aspirin and Wintergreen Oil

Symbolic equation

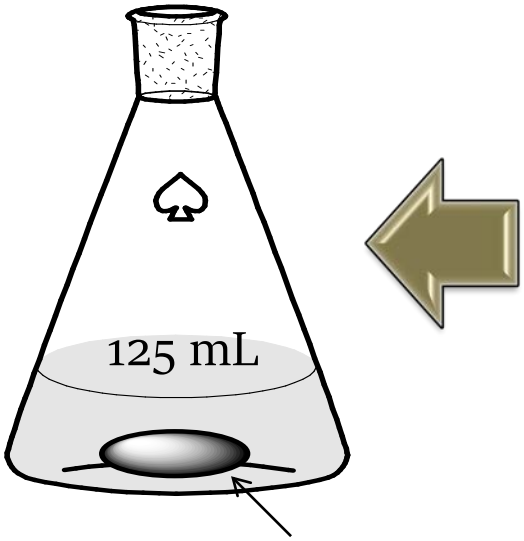




1. Mix the starting material and heat.

Set up a boiling water bath in a 400-mL beaker. Prepare about 100 mL of distilled water. Also set up an ice bath.

Measure about 2 g ($\pm$0.01g) of salicylic acid (Caution: This is a skin irritant) in a dry 125-mL Erlenmeyer flask. Cover the crystals with 4-5 mL of acetic anhydride is a severe eye irritant-avoid skin and eye contact) Swirl the flask to wet the salicylic acid crystals. Add 5 drops of conc H_2SO_4 (Caution: H_2SO_4 causes severe skin burns.) to the mixture and gently heat the flask in a boiling water bath for 5-10 minutes.



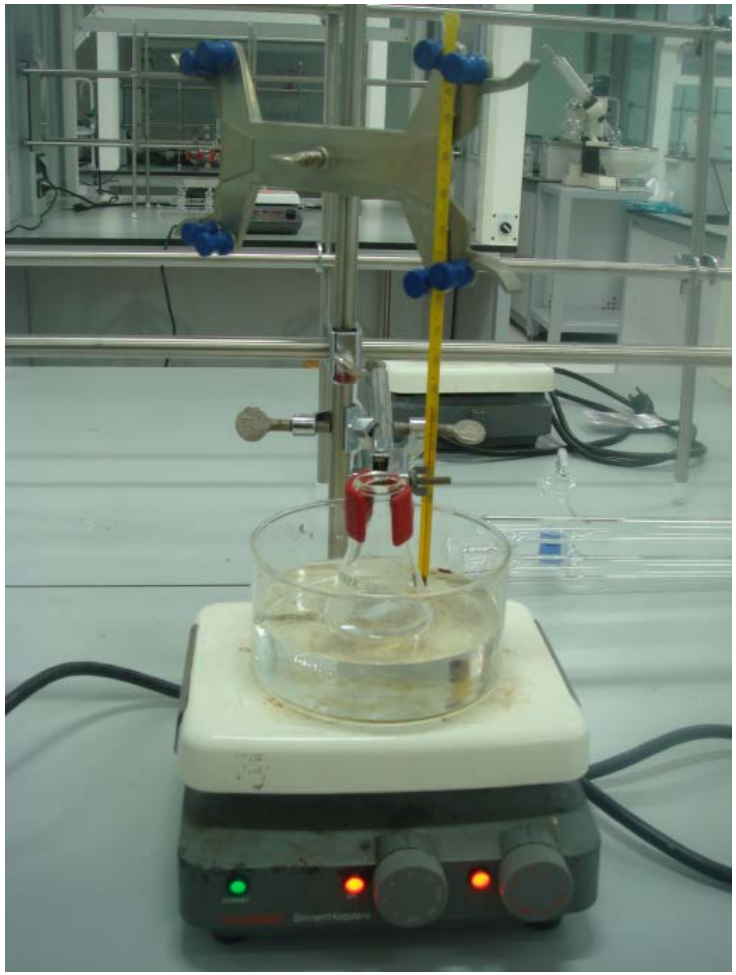
① Salicylic acid 2.0 g

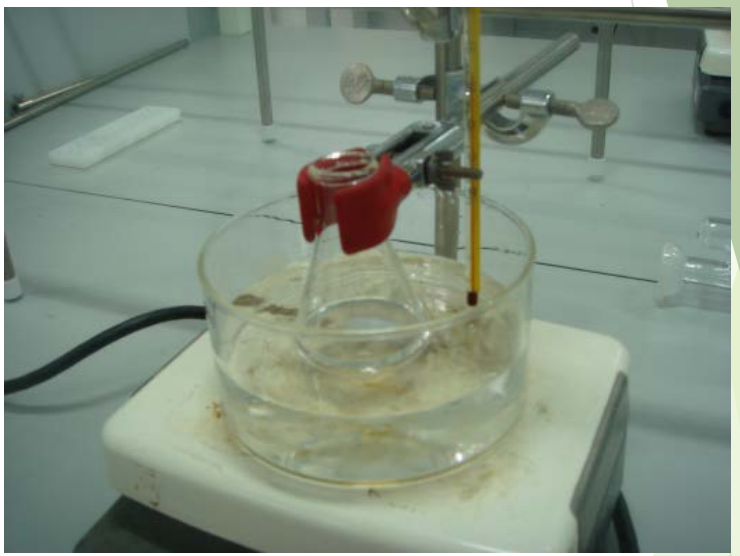
② Acetic anhydride 5.0 mL

(CAUTION ! Use it under the hood)

③ Sulfuric acid 5 drops

CAUTION! Acetic anhydride and concentrated H_2SO_4 can cause Severe burns if they come in contact with skin. If you get any of these reagents on you, immediately wash the area with copious amounts of water.





2. Cool to crystalline the aspiring.

Remove the flask from the hot water bath and, to the reaction mixture, add 10 mL of deionized **ice** *water to decompose any excess acetic anhydride. Chill the solution in an ice bath until crystals of aspirin no longer form, stirring occasionally to decompose residual acetic anhydride. If an "oil" appears instead of a solid, reheat the flask in the hot water bath until the oil disappears and again cool.*

3. Separate the solid aspiring from the solutions.

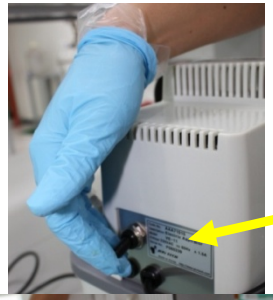
Set up a vacuum filtration apparatus and turn it on. Seal the filter paper with a water in the Buchner funnel. Decant the liquid from PART A.2 onto the filter paper; minimize any transfer of the solid aspirin. Some aspirin, however, may be inadvertently transferred to the filter; that's okay.

4. Filter, wash, and transfer the aspirin.

Add 15 mL of ice water to the flask, swirl, chill briefly, and decant onto the filter. Repeat until the transfer of the crystals to the vacuum filter is complete; maintain the vacuum to dry the crystals as best possible. Wash the aspirin crystals on the filter paper with 10 mL of ice water. Keep all of the filtrate until the aspirin has been transferred to the filter.

If aspirin forms in the filtrate, transfer this filtrate and aspirin to a beaker, chill in an ice bath, and vacuum filter as before, using a new piece of filter paper.

Tips! Stick the wet filter paper very closely on the funnel by suction before the filtration for good filtration and dryness of your product !!!



ON/OFF

5. Recrystallize the aspirin.

Transfer the crystals from the filter paper(s) to a 100-mL beaker. Add repetitive small volumes of ethanol (e.g. 3-mL volumes) to the aspirin until the crystals just dissolve (~10 mL is required). Warm the mixture in a 60°C water bath (Cation: No flame-use a hot plate or a hot water bath). Pour 5-10 mL of ~60°C water into the solution. If a solid forms, continue warming until the solid dissolves but do not boil. Add more water to dissolve if necessary.

Cover the beaker with a watchglass, remove it from the heat, and set it aside to cool slowly to room temperature. Then set the beaker in an ice bath.

6. How much did you prepare?

Vacuum filter the crystals on filter paper, the mass of which has been previously measured ($\pm$0.01 g). Wash the crystals with two 10-mL volumes of ice water. Place the filter paper and aspirin sample on a watchglass and allow them to air-dry. The time for air-drying the sample may require that it be left in your lab drawer until the next laboratory period.

Determine the mass of the dry filter paper and sample. Dispose of the filtrate as directed by your TA.

7. Correct for residual solubility.

The solubility of acetylsalicylic acid is 0.25 g per 100 mL water. Correcting for this inherent loss of product due to the wash water in PART A.6, calculate the percent yield.

$$\%\ yield = \frac{Actual\ yield\ (grams)}{Theoretical\ yield\ (grams)} \times 100$$

PART B. Melting Point of the Aspirin Sample (Vernier Melt Station)

2nd week

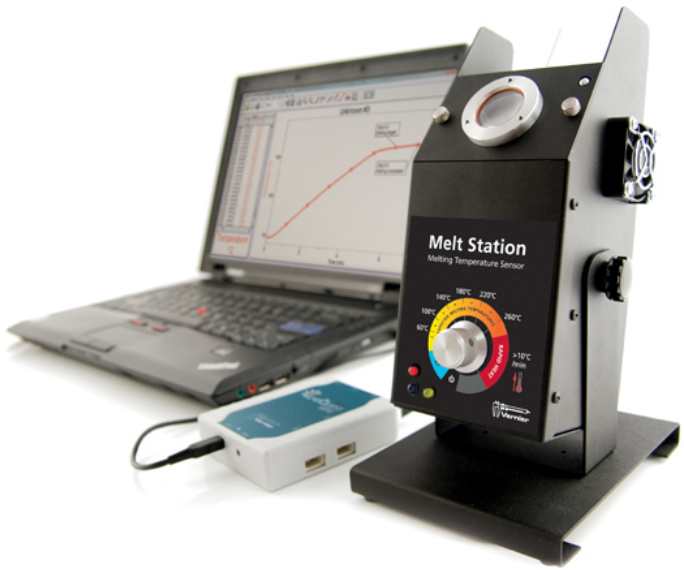
Example Procedure for Computer-Based Data Collection

1. Obtain and wear goggles.

2. 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.

3. Connect the Melt Station sensor cable to a LabQuest or computer interface.

4. Obtain a small amount of a solid organic compound. The solid should be in powder form. If it is not, use a mortar and pestle to carefully grind the solid to a powder.



: Off position

: Cooling fan on

: Rapid heating; heating rate at >10°C/min

5. Prepare a sample for melting.

a. Pack a capillary tube 3–4 mm (~1/8 inch) deep with your sample by inserting the open end into a small pile of the solid. A small amount of the solid will be pushed up into the tube.
b. Wipe off any loose solid that is on the outside of the capillary tube.
c. Tap the closed end of the capillary tube on the desk top to compress the sample into the closed end.
d. (optional) To further pack down the sample in the tube, drop the capillary tube (closed end down) down a section of glass tubing that has been set up for this purpose.
e. Carefully insert the capillary tube of solid into one of the three slots in the heating block of the Melt Station. You may rotate the Melt Station toward you slightly for a better look at the heating block.
f. Rotate the Melt Station up or down slightly to get the best view of the solid sample through the viewing lens.

6. Start the data-collection program, and then choose New from the File menu. You are now set
up to take melting temperature data for up to 20 minutes.

7. In the first trial, you will want to observe the melting process and make a *rough estimate* of the melting temperature of your sample. Do not worry if the heating rate is a bit too rapid, and the sample melts too quickly. To do this:

a. Start data collection.
b. On the Melt Station, turn the control knob to a setting of 180ºC. The red light will turn on indicating active heating.
c. Carefully observe your sample. If the solid begins to melt, click Mark to mark the temperature on your graph (or press the **D** key on the computer.) When the entire solid has completely melted, click Mark again. The two values marked on your graph describe the estimated melting temperature range of your substance.

d. If the solid does not melt by the time the temperature gets to 150ºC, turn the control knob to the 220ºC setting. Continue observing your sample, and if the sample begins to melt, mark the temperatures on the graph as previously described.
e. If the sample has not melted by the time the temperature gets to 190ºC, turn the knob to the Rapid Heat setting. When the sample finally begins to melt, mark the graph as previously indicated.
f. When you have determined the approximate melting temperature range for the sample, stop data collection. Store the run by tapping the File Cabinet icon in LabQuest, or choosing Store Latest Run from the Experiment menu in Logger *Pro*. Discard the capillary tube and sample as directed by your instructor.
g. On the Melt Station, turn the control knob to the Fan/Cooling setting to get ready for the next trial. The blue light will turn on indicating that the fan is cooling the Melt Station.

8. Now that you have a rough idea of the melting temperature, a more accurate determination can be made. Prepare a new sample in a capillary tube, as described in Step 5, to determine the melting temperature.

a. Start data collection.
b. On the Melt Station, turn the control knob to the Rapid Heat setting.
c. Carefully observe the temperature *vs*. time graph. When the temperature is within approximately 10°C of the lowest possible melting temperature of your sample, turn the control knob to a temperature setting corresponding to your expected melting temperature.
d. Carefully observe your sample. When the solid begins to melt, click Mark to mark the temperature on your graph. When the entire solid has completely melted, click Mark again. The two values marked on your graph describe the estimated melting temperature range of your substance. When you are finished with this step, stop data collection.
e. Store the run.
f. Discard the capillary tube and sample as directed by your instructor.
g. On the Melt Station, turn the control knob to the Fan/Cooling setting to get ready for the next trial.

9. At the end of the experiment, record the melting temperature range and turn the control knob on the Melt Station to Off.

10. 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.

PART C. Percent Acetylsalicylic acid in the Aspirin Sample

Three trials are to be completed in the analysis of the aspirin. Prepare three clean 250-mL Erlenmeyer flasks and determine the mass of three aspirin samples while occupying the balance. Obtain a 50-mL buret.

1. Prepare the aspirin sample for analysis.
   Assuming 100% purity of your aspirin sample, calculate the mass of aspirin that requires 20 mL of 0.1 M NaOH to reach the stoichiometric point. Weighing paper, measure the calculated mass ($\pm$0.001 g) of the aspirin you have just prepared and transfer it to the flask. Add 10 mL of 95% ethanol, followed by about 50 mL of deionized water, and swirl to dissolve the aspirin. Add 2 drops of phenolphthalein indicator. Repeat for trials 2 and 3.

2. Prepare the buret for titration. *Prepare a clean buret, rinse, and fill it with a standardized 0.1 M NaOH solution. Be sure that no air bubbles are present in the buret tip. After 10-15 seconds, read and record the volume, and the actual molar concentration of the NaOH solution.*

3. Titrate the sample. *Slowly add the NaOH solution from the buret to the dissolved aspirin sample until the endpoint is reached. The endpoint in the titration should be within one-half drop of a faint pink color. The color should persist for 30 seconds. Read and record the final volume of NaOH in the buret.*

DISPOSAL

Discard the solution in the Waste Organic Solvent container.