PREPARATION OF ASPIRIN

Objective

In this investigation, you will

- Prepare 2-acetyloxybenzoic acid, an ester, and
- Calculate the theoretical yield and percent yield.

EQUIPMENT

100-cm^3 beaker	2g 2-hydroxynenzoic acid (salicylic acid)
Paper towels	5 cm^3 acetic anhydride
Filter paper, 2 sheets	6 drops conc. H_2SO_4
Funnel	5 cm^3 distilled water
Ring stand	balance
Ring	thermometer
Hot plate	goggles
Hot water bath	apron
Ice	

PROCEDURE

CAUTION: *2-hydroxybenzoic acid and acetic anhydride are flammable and poisonous. Do not use near flames or heat. Acetic anhydride liquid and vapor is strong eye and skin irritants. Sulfuric acid is very corrosive. Wear goggles and an apron. Avoid skin contact. Rinse spills with plenty of water. Your product will contain contaminants; it is not safe for consumption*

PART A

1. Determine the mass of a clean, dry 100-cm^3 beaker. Place approximately 2.00 g of 2-hydroxybenzoic acid into the beaker. Record the mass in the table. Add 5 cm^3 of acetic anhydride and 5-6 drops of concentrated H_2SO_4. (The H_2SO_4 acts as a catalyst for the reaction.)
2. Swirl the contents of the beaker gently until a solution forms. Place the beaker into an 80°C water bath. Keep the contents of the beaker at 80°C for 12 to 15 minutes.
3. Remove the beaker from the hot water bath, and place it into an ice water bath. As the beaker cools, add 5 cm^3 of distilled water very slowly to the beaker. The water will react with the excess acetic anhydride to form acetic acid. Continue to cool the beaker and contents as you add 40 cm^3 of ice water to the products in the beaker. Crystals should begin to appear after about 1 minute. Allow the beaker to remain in the ice water bath for 5 minutes.
4. Set up a ring stand and ring to support a funnel for filtering. Place the filter paper in the funnel and transfer the crystals to the funnel for filtering and washing. Wash the crystals twice with 20 cm^3 of ice water.
5. After washing, unfold the filter paper and place it on top of several paper towels; blot to absorb the water from the filter paper. Allow the crystals to continue to air dry until the next lab period. Carefully transfer the crystals to a pre-massed piece of dry filter paper. Then mass the dry filter paper and crystals. Record your data in the table

PART B – *Testing the Aspirin*

Background Info: When the golden brown $FeCl_3$ is dispensed from a pipette onto white salicylic acid, a dark purple color will immediately appear. Aspirin (ASA) will not immediately produce a dramatic color change. However, ASA, which is not very soluble in water, will slowly react with water to produce salicylic acid and as a result of the slow hydrolysis reaction, a pale purple color

slowly appears.

1. To test for any unreacted salicylic acid, add a few crystals of salicylic acid to a TT. Dissolve the sample in 5 mL of water.
2. Add 1 drop of $FeCl_3$ solution. Note the color produced by the salicylic acid.
3. Repeat, using a few crystals of your aspiring instead of the salicylic acid. Does the aspirin contain any unreacted salicylic acid or is it pure?

CALCULATION

1. Calculate the theoretical yield of aspirin.
2. After the aspirin is dry weigh it on the filter paper, and calculate your percentage yield.
3. Calculate the theoretical yield of the aspirin. Calculate the percent yield.
4. Compare your % yield to the other groups who did the lab at the same time as you did.

DISCUSSION

1. What are the three main uses of aspirin?
2. Why do you think sulfuric acid was added to the reacting compounds?
3. Who discovered aspirin and what led to this discovery?
4. Acetylsalicylic acid “medicine” was developed in 1899. What tree did it come from? Which part of the tree was used for medicine? Why?

RESOURCES

CONCLUSION