Lab- Honors [30 pts] ***Name*** _____

Synthesis of Aspirin ***Lab Partner*** _____

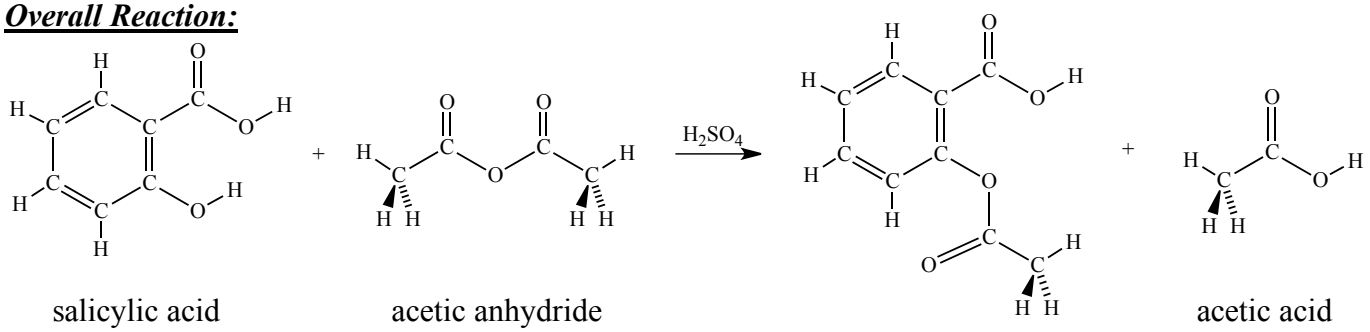
Period _____ ***Date*** _____

Introduction: The history of aspirin began on June 2, 1763, when Edward Stone, a British clergyman announced that the bark of a willow tree could reduce the feverish symptoms of malaria. A century later, a Scottish physician found that the extract of willow bark would relieve fever, reduce pain and reduce swelling. Soon after, organic chemists isolated and identified the active ingredient in willow bark as salicylic acid (from *salix*, Latin for willow tree). Salicylic acid could then be artificially produced in large quantities for medical use.

However, it was found that salicylic acid's medical uses were severely limited because of its acidic properties-- it causes severe irritation of the stomach lining. A breakthrough came when Felix Hofmann, a chemist at the German firm of Bayer, determined how to synthesize acetylsalicylic acid. Acetylsalicylic acid has all the same medicinal properties as salicylic acid, but it does not cause as much stomach irritation. Bayer called its new product "aspirin."

***Purpose*:** The purpose of this lab is to synthesize aspirin from salicylic acid (similar to how Hofmann did it!). You will carry out some common organic synthesis techniques (heating, filtration), determine your percent yield and test for the purity of your aspirin.

Overall Reaction:



salicylic acid + acetic anhydride → acetylsalicylic acid (aspirin) + acetic acid (vinegar)

Procedure:

1) Set up and begin heating a hot water bath. *Use a hot plate, large beaker & thermometer.* While heating, one person MUST watch the hot water at ALL times.
2) Mass out approximately **one gram** of salicylic acid. Record your **exact mass** of the salicylic acid.
3) Put the salicylic acid into a small 50 mL Erlenmeyer flask. Add 2 mL of acetic anhydride. Swirl.
4) Come to your teacher who will add 3 drops of conc. H_2SO_4. Swirl flask until dissolved.
5) When the hot water bath temperature is around 65°C, turn the hot plate off. Keeping the temperature between 70°C and 75°C, carefully put the flask into the hot water bath and secure tightly with a clamp. **Heat for 10 minutes.** (*Be Careful. Don't knock anything over. Acetic anhydride is FLAMMABLE!!)* Do not let the temperature of hot water bath get higher than 80°C. *If it gets too hot, it may cause the aspirin to decompose back to salicylic acid.*
6) After heating for 10 minutes, turn off the Bunsen burner, remove the flask and let it cool for 3 minutes. Get some beaker tongs and put the hot water beaker onto the lab bench to cool.
7) Add 10 mL of ***distilled*** water to the flask.
8) Cool the flask in an ice-water bath. After a few minutes you should start to see some white solid forming. If you don't, scratch the glass on the inside of the flask with a stirring rod to encourage the aspirin to crystallize. *(Be patient-- it will crystallize!)* Continue cooling the flask until there is complete crystallization. It may take about 20 minutes. If you get a relatively thick solid-liquid mixture, call your teacher over to check before moving on.
9) Put your names on a piece of filter paper with a **pencil**. Mass it out.
10) Collect your aspirin by filtering your mixture (ring stand, ring, funnel, filter paper, wash bottle, beaker). Use a wash bottle to wash all of the aspirin out of the flask and into the filter paper.
11) Put the filter paper/aspirin on a tray to dry overnight.

CLEAN UP: Wash out all glassware and put on racks to dry. Put everything else back where you got it.

DAY 2:

12) Mass out the dry filter paper/aspirin. Calculate the mass of aspirin you made.
13) **Test the purity of your aspirin**: *(Is your aspirin product contaminated by salicylic acid?)*
    a) Get three small test tubes and add about 5 mL of water to each.
    b) Add a few crystals of salicylic acid to one tt, commercially made aspirin in another tt, and your aspirin product in the third tt. (*Use about same amount in each tt)*
    c) Add ~5 drops of $Fe(NO_3)_3$ solution to each test tube and note the color in each. *(If salicylic acid is present, a Fe^{+3}-salicylic acid complex will form. This complex has a definite violet color.)*
14) Put the remaining amount of your aspirin in a labeled vial (names, period). Later this year, we may test the purity of your aspirin again using a more precise method.

Data: [2 pts]	**Purity tests**:
Mass of salicylic acid = _____	color of solution with salicylic acid =_____
Mass of filter paper = _____	color of solution with commercial aspirin =_____
Mass of filter paper/aspirin = _____	color of solution with your aspirin = _____
Mass of aspirin = _____	

Complete the rest of the lab on a separate sheet of paper.

Calculations: [8 pts] Show all work!! Show all units and WATCH your SIG FIGS!!

Helpful information: *MM of salicylic acid = 138 g/mole; MM of acetic anhydride = 102 g/mole*
MM of acetylsalicylic acid = 180 g/mole; density of acetic anhydride = 1.08 g/mL

a) [3 pts] What is the limiting reactant in this reaction?
   Hint: Calculate the moles of salicylic acid used and calculate the moles of acetic anhydride used.
b) [3 pts] Calculate the theoretical yield of aspirin. (In other words, calculate the grams of aspirin that should have been produced assuming that all of the limiting reactant reacted.) *Show each step clearly-- label values with the name of substance. Don't forget to use the balanced equation on the front*.
c) [2 pts] Calculate your percent yield of aspirin. (Show work.*)*

Discussion: [10 pts] *Discuss in two, well written, organized paragraphs.*

- Report your percent yield. Did you get a good yield of aspirin (70% or better is pretty good)? What possible sources of loss were there and what could you have done to improve your yield (answer this question even if you had a good yield)?
- Was your aspirin relatively pure or was it contaminated by salicylic acid (state experimental support for your decision)? Even if your aspirin was relatively pure, what errors could account for some impurity?

Post Lab Questions: [10 pts]

1) [3 pts] Write in all **significant** partial charges where appropriate on all of the atoms in the structures on first page. (Just fill in δ^+, δ^- symbols above the appropriate atoms on the first page of the lab handout.)
2) [1 pt] Where is salicylic acid naturally found? *(read intro of lab)*
3) [2 pt] Why is it better to take acetylsalicylic acid to relieve a fever than to take salicylic acid? *(Read intro)*
4) [2 pts] When trying to crystallize your product from solution, why was it important for you to put your flask in an ice bath? *Hint: What temperature water can dissolve more sugar? hot water or cold water*
5) [2 pts] In the procedure I warned you not to warm your solution too hot (over 80°C) because the aspirin might decompose back to salicylic acid at this high temperature. If it did decompose, it would go by this reaction: **aspirin + H_2O ⟶ salicylic acid + acetic acid**

   Similarly, this reaction can also take place when aspirin reacts with water from the air over time. Thus, sometimes, one can smell some acetic acid (vinegar) in an old aspirin bottle. Why would it be harmful for you to ingest this old aspirin?