(H) Chemistry **Name:**
Preparation of Aspirin Lab **Blk:** **Date:**
(% yield) **Lab#**_____

Introduction:

In 1851, Charles Gerhardt, a chemist, began to research a means to administer pain relief which would be more efficient than chewing willow tree bark and less irritating to the stomach and gums. He knew that pure salicylic acid killed pain but the side effects were a deterrent. After much research and 2 years of experimentation, he discovered a process which used acetic anhydride and created acetylsalicylic acid (ASA) the precursor to the modern day aspirin. It took 40 years for another scientist, Felix Hoffman, to devise a process based on Gerhardt's work, which was practical for producing reasonable quantities of the pain killer.

Aspirin is prepared in the following basic reaction:

$$C_7H_6O_3 + C_4H_6O_3 \xrightarrow{H_2SO_4} C_9H_8O_4 + HC_2H_3O_2$$

salicylic acid + *acetic anhydride* → *acetylsalicylic acid* + *acetic acid*

Your % yield depends on how carefully you follow the procedure and certain other experimental factors.

Procedure:

1. Set up a water bath using a 250 mL beaker (half full of water) on a ring stand and ceramic-centered wire gauze. Heat to a constant temperature of ~75° C (<u>not higher than 80° C</u>). This will require a very soft, low flame on your Bunsen burner.

2. While adjusting the water bath, mass a clean, dry 100 mL beaker and **add 1.00 g of salicylic acid** to the beaker. Record these masses.

3. Bring your beaker to the teacher demo table where the instructor will pipet **3.0 mL of acetic anhydride** into the beaker. Immediately, **add 3 drops of the catalyst concentrated sulfuric acid** (using an eye dropper provided by the instructor).

4. Place the 100 mL beaker carefully into the 250 mL beaker and use the thermometer to stabilize the apparatus. Do this only if the temperature has stabilized at a fairly constant 75° C.

5. Heat the reaction chamber (100 mL beaker in water bath) for 15 min.

6. Use the crucible tongs to carefully remove the reaction chamber from the water bath. **Slowly, and immediately add 20 drops of distilled water** to the solution to decompose any unreacted acetic anhydride.

7. While allowing the reaction beaker (100 mL) to cool, empty the warm water bath using beaker tongs. After this beaker (250 mL) has cooled a bit, fill it half full of ice water to create an ice bath used to facilitate the formation of aspirin crystals.

8. Wait only a couple of minutes for the reaction beaker to cool somewhat, then add an additional 15 mL of distilled water to the reaction beaker to complete the anhydride decomposition. **Immediately, place the 100 mL reaction beaker into the ice bath** to re-crystallize the aspirin. The longer this can sit in the ice the better the product recovery is. (you may need to just barely touch the inside of the beaker with a glass rod to start the crystal formation if it doesn't start fairly quickly – swirling gently helps).

9. While waiting on crystal formation, **mark (in pencil) and weigh a piece of filter paper.** Record this mass. Set up a funnel in a 100 mL graduated cylinder to catch the residue. (you may also use the ring stand, but students in the next class may need to move your apparatus – take your own risks).

10. When crystallization appears complete (it should be a mostly stiff, moist-looking crystal sludge), use the glass rod to very carefully scoop the crystals into the funnel for filtration. Cold ice water (~5-10 mL at most) can be used to help swirl the crystals and carry them to the beaker lip in order to help get the maximum crystal yield. Be careful not to over-run your filter or funnel.

11. This filter can be brought to the instructor for drying. Mass the final filter paper + aspirin during the next lab period. Complete calculations.

Data: (show ALL set-ups for work with labels)

A. Mass of empty 100 mL beaker _____

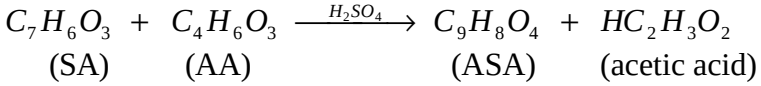
B. Mass of beaker + salicylic acid _____

C. Mass of salicylic acid ("B" – "A") _____

D. Mass of filter paper _____

E. Mass of filter paper + aspirin _____

F. Mass of aspirin ("E" – "D") _____
(actual yield)



Using this equation, calculate the theoretical yield and then your % yield.

Molar mass of acetic anhydride _____

Molar mass of salicylic acid _____

Molar mass of aspirin _____

moles of salicylic acid added to beaker _____

if density of acetic anhydride is 1.05 g/mL, what was the mass of 3.0 mL of acetic anhydride added?

_____

moles of acetic anhydride added to reaction _____

According to the mole ratios in the given reaction, what is the limiting reagent? Show rationale

_____

What is the theoretical yield from the balanced equation? _____

What was your percent yield? _____

Conclusions:

In complete sentences, identify any possible sources of error which would/could affect your % yield. Please be specific (don't say it would create an error – what kind of error???)