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Lab work: industrial organic chemistry
Specialty: Process Engineering
Level: 2nd year

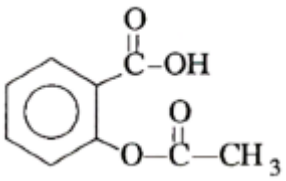
Experiment 01: SYNTHESIS OF ASPIRIN

I. Objectives:

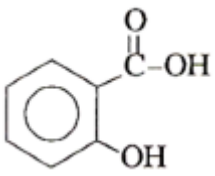
- Synthesize acetylsalicylic acid (aspirin) by carrying out a simple organic reaction,
- Separate the product from the reaction mixture by vacuum filtration,
- Purify the product by recrystallization,
- Perform a chemical test to identify the change in functional group from reactant to product,
- Determine the success of the synthesis by calculating the percentage yield of the product.

I. Introduction:

Aspirin is one of the most widely used medications in the world. It is employed as an analgesic (pain relief), an anti-pyretic (fever control) and an anti-inflammatory. More recently, studies have indicated that daily intake of small doses of aspirin can lower the risk of heart attack and stroke in high-risk patients.



Acetylsalicylic acid
(Aspirin)



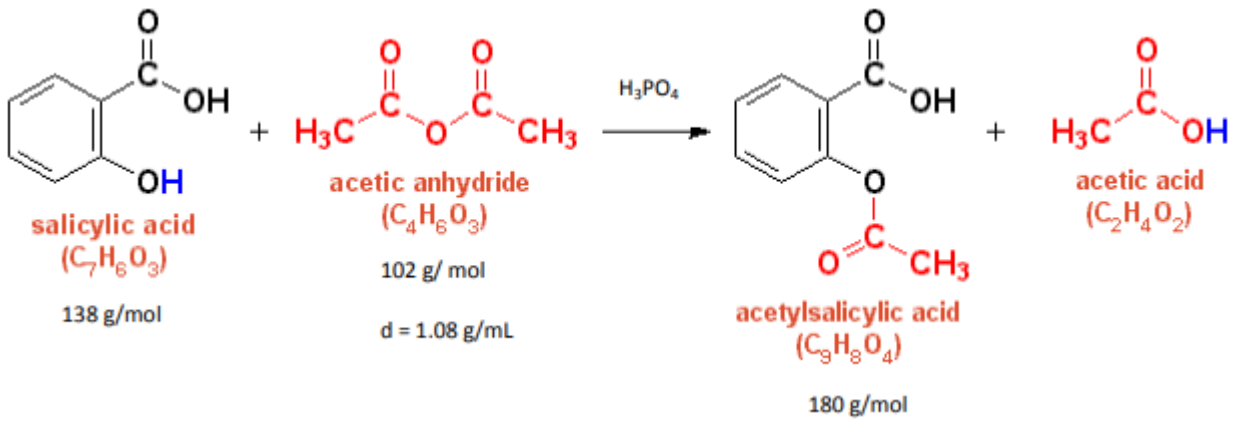
Salicylic acid

The history of aspirin and its precursor dates back to ancient times. Documents attributed to Hippocrates, the father of modern medicine, from the 4th century B.C. refer to the alleviation of pain by chewing on the bark of a willow tree or ingesting a powder made from the bark and leaves of the willow. This remedy was passed on from generation to generation.

II. Priciple:

To prepare aspirin, salicylic acid is reacted with an excess of acetic anhydride. A small amount of a strong acid is used as a catalyst which speeds up the reaction. The excess acetic acid will be

quenched with the addition of water. The aspirin product is not very soluble in water so the aspirin product will precipitate when water is added. The synthesis reaction of aspirin is shown below:



III. Equipment and Reagents:

- Salicylic acid	- Buret clamp	- Burner
- Acetic anhydride	- Stand with iron ring	- Distilled water
- Sulfuric acid	- Wire gauze	- Ice bath
- Phenolphthalein	- Beaker of tap water	-filter paper
- Reflux heating apparatus	- Thermometer 0 to 100 °C.	- Heating magnetic stirrer

IV. Experimental Procedure :

a)- Aspirin Synthesis :

Step	Procedure
1	Measure out 5 g of salicylic acid (SA) and place the SA in to 250-mL Round-bottom flasks.
2	Add, with continuous stirring, 7 mL of acetic anhydride (fume hood) followed by 4 to 5 drops of concentrated sulfuric acid.
3	Clamp the flask in a beaker of tap water supported on a ring stand over a burner flame. Stir if needed to dissolve the salicylic acid. Heat the water to boiling, and shut off the flame. Keep the flask in the hot water bath for 10 more minutes.
4	While the flask is still in the water bath, slowly add 2 mL of distilled water to the flask to decompose any excess acetic anhydride.

5	After a minute, remove the flask from the water bath and add 20 mL of distilled water. Let the flask cool to room temperature. As the solution cools, crystals of aspirin will appear. Cool the solution further by placing the reaction flask in an ice bath. Chill 5-10 mL of distilled water in a separate container.
6	Set up a Büchner funnel on a vacuum flask connected to a water aspirator. Place the filter paper in the funnel and moisten with distilled water from a squirt bottle. Turn on the aspirator and transfer the aspirin slurry into the funnel. Wash the crystals with 5 mL of the cold water.
7	Transfer the filter paper and aspirin to a pre-weighed watch glass and allow to air dry in your locker until the next lab period.
8	It is safe to discard of the filtrate down the sink with water.

b)- Recrystallization of the Product:

Step	**Procedure**
1	Transfer the impure aspirin from the Büchner funnel to a 150-mL beaker. Add 4 mL of 95% ethanol and warm the flask on a hot plate until all of the solid dissolves.
2	Immediately remove the flask from the heat and slowly add 20 mL of cold water.
3	Crystals should form. Chill this solution in an ice-water bath, and collect the crystals using vacuum filtration.

c) Yield of Purified Aspirin:

Once your aspirin appears to be dry, transfer it to the watchglass. Determine and record the mass of the watchglass and dried aspirin.

$$\%\ Purity = \frac{actual\ moles\ of\ acid}{theoretical\ moles\ of\ aspirin} \times 100$$

d)- Titration Analysis:

Step	Procedure
1	Accurately weigh between 0.5 g of the aspirin product into a 125 mL Erlenmeyer flask. Add 15 mL of 95 % ethanol and swirl to dissolve. Add 2 drops of phenolphthalein indicator to the flask.
2	Record the exact concentration of the standard 0.1 M NaOH solution. Fill a buret with the standard NaOH solution and record the initial volume. Titrate the sample until a faint pink end point is reached. The pink color should last for at least 30 seconds after swirling.
3	Repeat the titration with 0.5 g of a crushed aspirin tablet. You may need to use a mortar and pestle to crush the tablet.

e)- Iron (III) Chloride Test for Phenols:

Step	Procedure
1	Place a match head-sized quantity of the pure aspirin into a medium test tube. Into a second medium test tube place a match head-sized quantity of salicylic acid.
2	Add 5 mL of distilled water to the tubes containing the aspirin and the salicylic acid, and to an empty tube. This third tube will be used as a control for color comparison. Agitate the tubes to dissolve the solids as completely as possible. Add 2 to 3 drops of 0.2 M $FeCl_3$ solution to each tube and shake. An orange to purple color is indicative of the presence of the phenol group of salicylic acid. Record your observations.

Questions:

1. Provide the semi-developed formulas of salicylic acid, acetic anhydride, and aspirin.
2. Write the overall equation for the reaction corresponding to the synthesis of acetylsalicylic acid.
3. What is the principle of recrystallization?
4. What is the effective yield of this synthesis?
5. What do you conclude from this experiment?