

Aspirin Synthesis and Analysis

Pre-lab: Pages 305 – 306

Qns: 1 to 8, 11, 12

Pre-lab: Page 313, Qns. 4 - 8

Post-Lab: Page 307-308, Qns. 1,2, 3b, 4, 5

Answer these questions in detail

Experimental Aims

- To prepare a sample of aspirin (organic synthesis).
- Part B will NOT be performed.
- Calculate the purity of a commercial sample of aspirin through titration

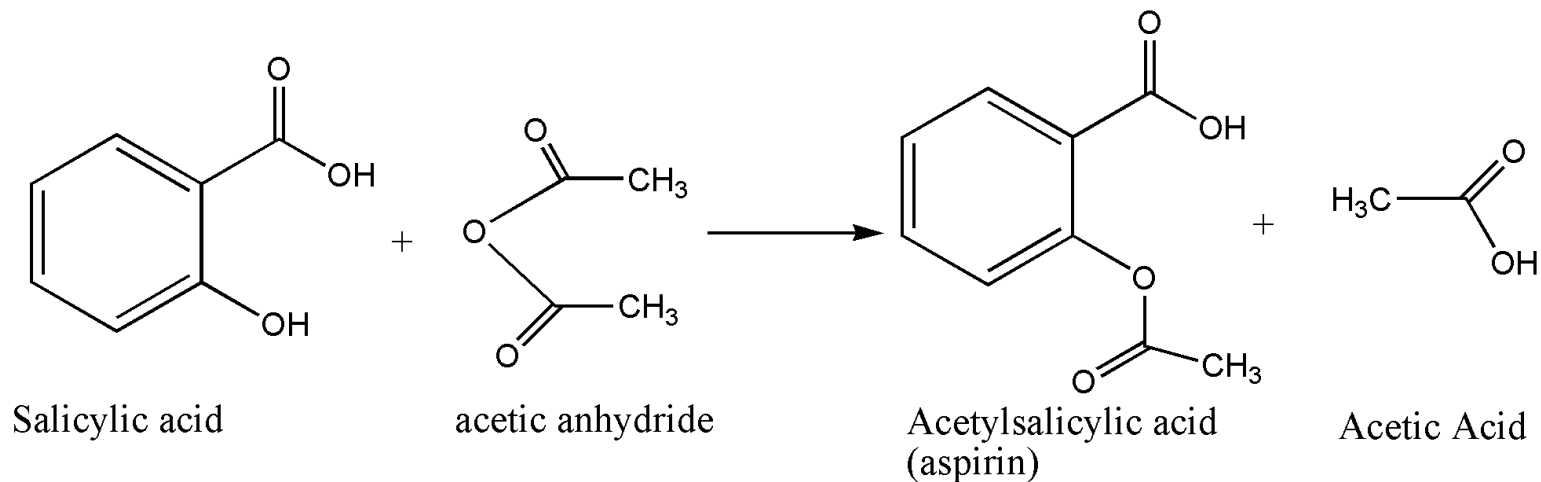
Esterification

- The synthesis of aspirin is an esterification reaction.
- Esterification is the general name for a chemical reaction in which two chemicals (typically an alcohol and an acid) form an ester as the reaction product (aspirin).
- Esterification is a reversible reaction.

Esters

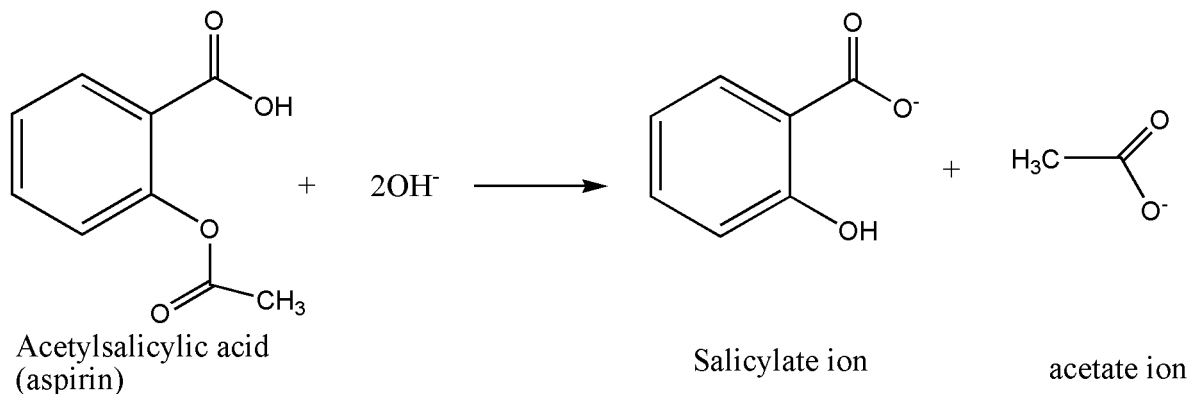
- Esters are derivatives of organic acids
- Have the functional group: $\text{RC}(=\text{O})(\text{OR})$
- They have pleasant, fruit-like odors.

Aspirin



- 2 g of salicylic acid and ~ 5 mls of acetic anhydride are added together in the hood.
- H_2SO_4 is added

Why add H₂SO₄?



- Acid is needed to keep the aspirin from being hydrolyzed (as shown above)

Experimental Procedures

- After mixing the reactants, the solution is heated for 5 – 10 minutes.
- On cooling add D.I water.
- This is to decompose any acetic anhydride that might be present

- Anhydride means without water
- Acetic anhydride is an acid anhydride. This means that when water is added to it, it will form an acidic solution.
- Acetic anhydride + water = Acetic acid

Crystallizing the aspirin

- This will be done in the same way as in the alum lab.
- If crystals are slow to grow:
 - Reduce the volume
 - Scratch the surface of the beaker
- Filter the crystals using a vacuum filtration

Testing the solid

- To test for unreacted salicylic acid, in a test tube dissolve a few crystals of your crude aspirin in water.
- Add a drop of 1% FeCl_3 .
- Note the color.
- Do the same reaction with a sample of salicylic acid.

FeCl₃ test

- Iron (III) Chloride is a test for phenol.
- A phenol is an OH group attached to a phenyl group.
- Phenols form colored complexes with iron(III) chloride
- A positive test (colored solution) on your aspirin sample indicates unreacted salicylic acid.

Recrystallize the product

- Recrystallization is a laboratory technique used to purify solids based on their different solubilities.
- A small amount of solvent is added to a flask containing an impure solid. The contents of the flask are heated until the solid dissolves.
- Next, the solution is cooled.
- A more pure solid precipitates, leaving impurities dissolved in the solvent.
- Vacuum filtration is used to isolate the crystals. The waste solution is discarded.

Recrystallization Steps

1. Add a small quantity of appropriate solvent to an impure solid.
2. Apply heat to dissolve the solid.
3. Cool the solution to crystallize the product.
4. Use vacuum filtration to isolate and dry the purified solid.

Yield

- Once your sample has dried, obtain its mass.
- Based on the mass of salicylic acid used calculate your theoretical and percent yield.
- If your yield is over 100%.. Explain why.

Safety Information

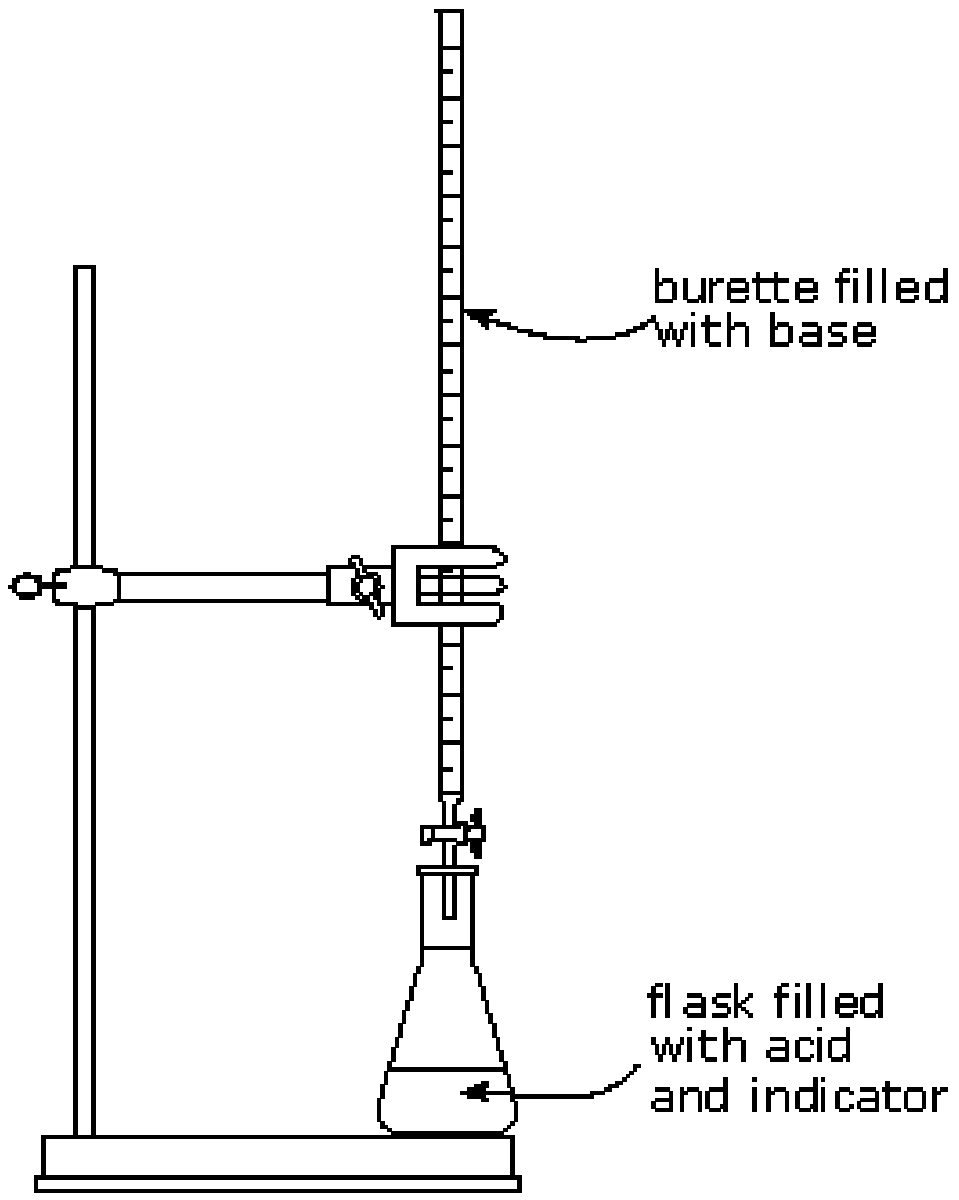
- Salicylic acid is a skin irritant
- H_2SO_4 – strong acid
- Acetic anhydride – lachrymator
- Recrystallizing from ethanol – do not have an open flame

Commercial Aspirin Purity

- Titration
- Need reproducible results
 - 2 trials are performed (3 titrations)
- Accuracy is important

Procedure

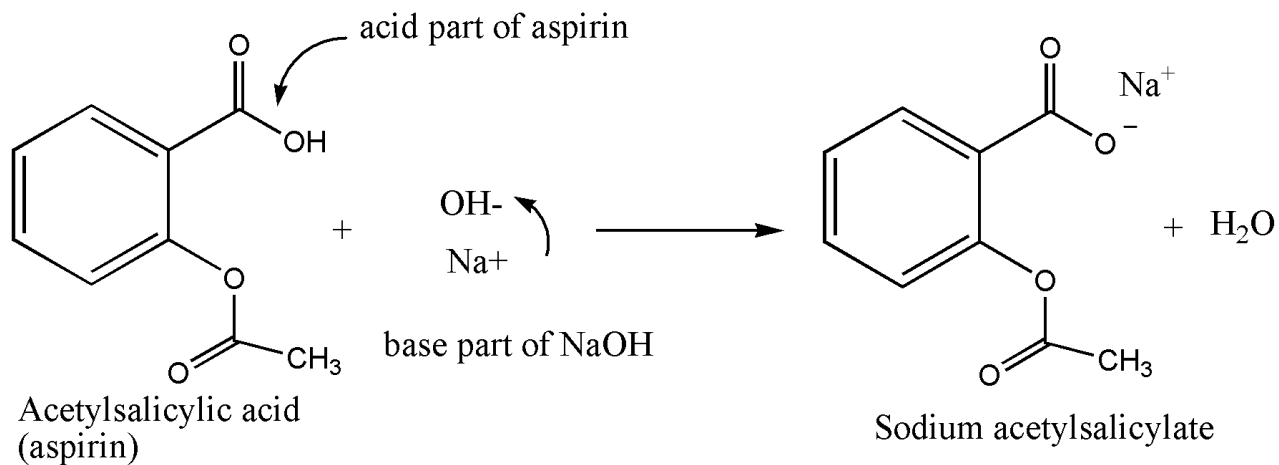
- The aspirin sample) will be titrated with NaOH
- You will be given the concentration of your NaOH.
- Shake up your NaOH, if your solution is not well mixed your reaction will not work



Procedure

- Take one aspirin pill
- It needs to be crushed and then weighed
- Use your concentration of NaOH from last week.
- Calculate approx. how many moles are present in 20 mL of your NaOH (this is a convenient volume to titrate)
- You can then estimate how much aspirin you will need to weigh

Aspirin Analysis



- 1 mole of aspirin reacts with 1 mole of NaOH
- Like KHP + NaOH. This is an acid-base reaction

- The aspirin will need to be dissolved in an ethanol / water mix
- The burets *should* not need to be cleaned
- Perform the analysis while your aspirin is crystallizing
- Titrate your aspirin 2 –3 times
- Accuracy is important

Calculations



- 1 mole of aspirin reacts with 1 mole of NaOH
- The concentration of NaOH is known
- The volume of NaOH needed to react with the aspirin will be known (vol dispensed from buret)
- Calculate moles of NaOH
- Moles of NaOH = Moles of acid

- Molecular weight of acetyl salicylic acid is known.
- Using moles calculated, calculate theoretical mass of acetyl salicylic acid
- Compare with actual mass of aspirin measured
- Determine % purity of the commercial sample