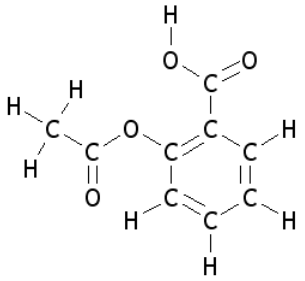
Lab: Titration of Aspirin

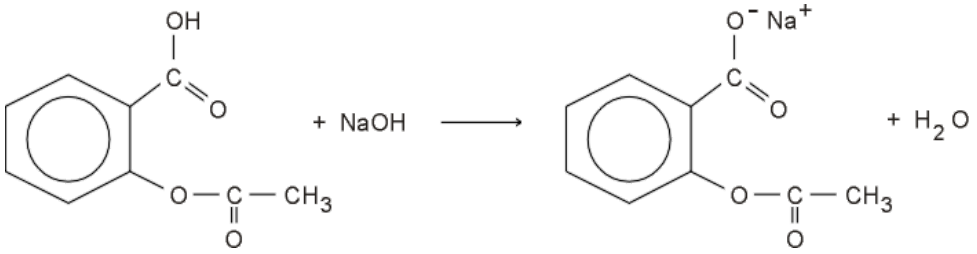
Objective: To determine the mass of aspirin in a commercially available aspirin tablet via titration.

Introduction:
The chemical name for aspirin is acetylsalicylic acid, which has the structure below.



Acetylsalicylic acid ($C_9H_8O_4$)

In this lab, you will dissolve an aspirin tablet in ethanol (since it does not dissolve easily in water). You will then use a buret to titrate the aspirin (i.e. acetylsalicylic acid) with sodium hydroxide (a strong base). The acid-base neutralization reaction is shown below using structural formulas, condensed formulas, and chemical names.



$$CH_3COOC_6H_4COOH + NaOH \rightarrow CH_3COOC_6H_4COO^-Na^+ + H_2O$$

Acetylsalicylic acid + sodium hydroxide → sodium acetylsalicylate + water

Phenolpthalein, an acid-base indicator that turns from clear to pink in the pH range 8.0-9.2, will be used to signal the point at which the added base has neutralized the acetylsalicylic acid.

Materials:

~ 0.1 mol dm^{-3} *standardized* sodium hydroxide solution
(actual concentration = _____ + ____ mol dm^{-3})* obtain from instructor
phenolphthalein indicator solution
ethanol (25 cm^3)
aspirin tablet
graduated cylinders (100-cm^3 and 10-cm^3)

Erlenmeyer flask (250 cm^3)
Small glass funnel
50 cm^3 buret
buret clamp
Wash bottle with distilled water

Safety: NaOH is corrosive. Handle with care. In case of contact with skin, rinse the area with large amounts of water and notify your instructor. Wear goggles at all times in the laboratory.

Procedure:

A buret is marked in milliliters like a graduated cylinder, but buret markings show **0 mL** at the top, and the numbers increase as you go down the buret. The stopcock controls the liquid flow. It is ***open*** when ***parallel*** to the length of the buret and ***closed*** when ***perpendicular*** to the length of the buret.

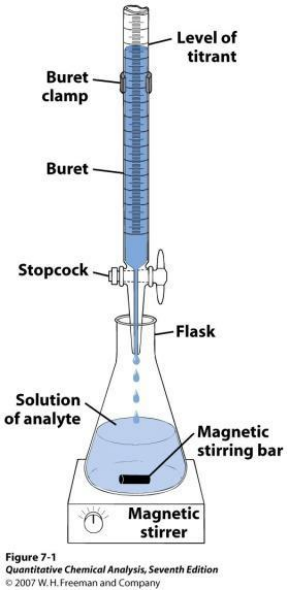


Figure 7-1
Quantitative Chemical Analysis, Seventh Edition

Conditioning the buret

1. Close the stopcock and add about 5 mL of the ***titrant*** (the solution to be used into the buret).
2. Tilt the buret sideways and roll the barrel to completely rinse the inner walls of the buret.
3. Drain the solution through the buret tip to insure the tip is also conditioned.
4. Repeat this step at least twice to be sure all interior surfaces are rinsed with titrant.

Filling the buret

1. Close the stopcock.
2. Use a clean funnel to fill the buret with titrant just above the "0" mark. Do NOT pour over your head. Place a container under the buret tip, and open the stopcock briefly to fill the buret tip with solution, leaving no air bubbles, and to get the level of meniscus to fall within the markings of the buret. Note: ***The initial level of titrant does NOT need to be exactly at 0.00 mL*** as the initial level of liquid will be recorded and subtracted from the final volume to determine the volume delivered.

Preparation of Aspirin

1. Obtain a tablet of commercially available aspirin, weigh it, and place it into a 250-mL Erlenmeyer flask. Record the brand name.
2. Add about 25 ml of distilled H_2O. to the flask. Allow the tablets to stand for a few minutes, and then tap the tablet using a glass stirring rod. The tablet may begin to break down.
3. Add about 25 mL ethanol to the flask to help the aspirin dissolve. Break up the remainder of the tablet with the stirring rod and swirl the flask until no pieces of the tablet remain. The solution may remain cloudy due to the insoluble starch binder used to hold the tablet together.
4. Add 3-4 drops of phenolphthalein indicator.

Titration of Aspirin

1. Record the initial buret reading.
2. In the box to the right, write the estimated volume of NaOH solution (V_{calc}) you calculated in the Pre-Lab.

V_{calc} = _____ cm^3

3. *Slowly* titrate the aspirin with the NaOH solution until a faint pink endpoint that persists after swirling. Use this to determine when your titration will approach the endpoint. Near this volume, a single drop of NaOH will cause the indicator to change color.
4. Open the stopcock and allow about 2 mL to drain from the buret. Close the stopcock and swirl the contents of the flask thoroughly.
5. Continue this procedure until you have delivered about 5 mL less than your V_{calc}. Add this point, begin adding **1 mL or less** of titrant at a time until you see the color of the indicator begin to persist while swirling. If any drops of titrant get stuck on the inside of the flask, you may use the wash bottle of distilled water to wash them down into the solution.
6. Now add NaOH dropwise until you reach a *faint pink* endpoint that persists upon swirling.
7. Record the final buret reading at the endpoint.
8. If time allows, repeat the titration with a second aspirin tablet.

Cleaning the buret: Most breakage occurs during washing, since burets do ***NOT*** fit under the faucet.

1. Empty the buret and dispose of the sodium hydroxide according to instructions.
2. With the buret over the sink and the stopcock open, use a beaker to pour tap water into the buret and let it drain out the tip.
3. Rinse with several portions of tap water, allowing some tap water to run through the tip.
4. Do a final rinse with small portions of distilled water, allowing the water to run through the tip, then clamp the buret upside down to dry.

Waste Disposal:

- Empty any excess sodium hydroxide solution into the waste beaker on the back lab bench.
- Your titrated aspirin solution should be emptied into the container in the fume hood.

Report:

Use the results of your titration(s) to calculate the **mass of aspirin (acetylsalicylic acid) in the aspirin tablet**.

Write a DCP report for the lab. This will be counted as an official IB Internal Assessment grade. Thus, your instructor will not assist you in making any calculations.