Determination of amount and percentage of aspirin from given sample of tablet

Introduction:

Aspirin (acetyl salicylic acid) tablets are prescribed to reduce headaches, pain, common cold, inflammation, etc. It is on the World Health Organization's List of Essential Medicines. The global annual consumption of aspirin is about 40,000 tons*. It is important to estimate the actual percentage of aspirin in the specified tablet for several purposes like to confirm whether the tablet sample contains the prescribed amount or whether the amount has varied few days after production of tablet. The structure of aspirin is shown below and its IUPAC name is 2-acetoxybenzoic acid.

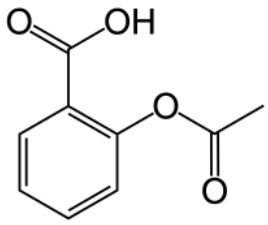


Figure 1: Aspirin

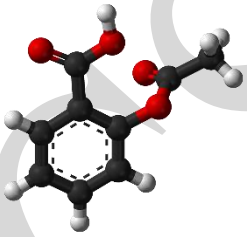


Figure 2: Aspirin-3D ball and stick model

Figure 3: Commercially available aspirin tablet

Photo credit
Figure 1- https://en.wikipedia.org/wiki/Aspirin#/media/File:Aspirin-skeletal.svg
Figure 2- https://en.wikipedia.org/wiki/Aspirin#/media/File:Aspirin-B-3D-balls.png
*Reference: https://en.wikipedia.org/wiki/Aspirin

In this experiment, we will be determine the percentage of aspirin in the supplied sample of the tablet using volumetric titration. You are aware that quantitative reaction with the titrant is basis of any titration. The current titration is an acid-base titration and we will add excess of NaOH to the aspirin tablet sample. We will be performing two titrations, namely, blank titration and back titration in the current experiment.

For blank titration, in the current experiment, the NaOH (to be added to aspirin sample) will be taken separately and after appropriate dilution, a fixed volume of diluted NaOH will be titrated with standard HCl solution.

Aspirin is first allowed to react with excess NaOH. The solution is appropriately diluted. Then the fixed volume of sample solution is titrated against standard HCl. Phenol-red or phenolphthalein can be used as an indicator.

Prelab Questions:

1. Consider the structure of aspirin in Figure 1. If suppose, water is added to small amount of aspirin tablet powder (please ignore other components like binders present in tablet) and is kept aside for 30 mins. Write the balanced chemical equation for the likely reaction that will happen.
2. If instead of water, aq NaOH solution is added to another aspirin tablet sample, what is likely reaction that will happen and write its balanced chemical equation.
3. Show appropriate calculations for preparation of the following solutions

a) 100 mL of 0.5 M NaOH (NaOH – molecular mass- 40 g)

b) 250 mL of 0.05 M HCl from concentrated HCl (Molarity =11.3 M)

4. In your opinion, when the back titration will be used in this experiment, what does it suggest about the chemical reaction between aspirin and NaOH?

Lab experiment

Objectives:

1) To determine amount of aspirin using hydrolysis reaction in basic medium.
2) To develop deeper understanding about back and blank titration techniques

Equipment and glassware:

Item	Quantity	Item	Quantity
Conical flask (100 mL)	2	Measuring cylinders (10mL)	1
Beaker (100mL)	1	Volumetric flask (100mL)	2
Test tube	1	Glass rod	1
Gloves		Boiling water bath	
Mortar and Pestle	1		

At your Work Bench

Chemicals	Quantity	Placed in
Aspirin Tablets	10 Nos. (75mg each)	
0.05 M HCl 	250 mL	beaker
0.5 M NaOH 	100 mL	beaker
Phenolphthalein 	few drops	drop bottle
Potassium hydrogen phthalate (KHP)	1.02 g	

It is helpful if you are familiar with the chemical hazard symbols. Such awareness gives a better understanding regarding handling and disposal of the chemicals you are using

 Flammable

 Harmful Irritant

 Corrosive

 Carcinogenic

Procedure:

Titration of NaOH (Blank titration)

1) Take 10 mL supplied NaOH solution with help of burette in a 100 mL standard measuring flask. Dilute this solution up to the mark with distilled water.
2) Take 10 mL diluted solution in a 100 mL conical flask, and 2-3 drops of phenolphthalein indicator. You will observe the pink colour in the solution.
3) Fill burette with standardized HCl solution, remove air bubble from the jet and adjust zero mark.
4) Titrate the NaOH solution with standardized HCl till pink colour just disappears. Note this reading in answer sheet and repeat the titration at least two more times (this is your '**X**' reading).

Titration of sample (Back Titration)

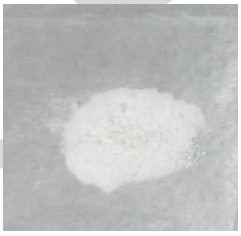
1) You are given 0.20 g of aspirin tablet sample. [Please Note The mass of powder is indicated on the vial. The way the sample was powdered – 6 tablet from a strip where grinded together and the sample given to you in the vial is weighed from this bulk powder. Mass of total powder (after grinding) = 0.360 g
2) Transfer the entire powder in a 100 mL conical flask and add 10 mL NaOH solution with the help burette.
3) Boil the mixture on a water bath for 5 minutes. Cool the solution after removing it from water bath (**Caution**: Handle the hot solution with care!!). *
4) Then dilute this solution in a 100 mL standard measuring flask using distilled water.
5) Pipette out 10 mL diluted solution in a 100 mL conical flask. Add 2 drops of phenolphthalein indicator and titrate with standardized HCl solution
6) Note the reading in the answer sheet and perform at least two more titrations in similar manner (this is your '**Y**' reading).

*- If the solution has undissolved sample (mostly binder) please filter the solution before diluting it.

Aspirin tablet sample preparation

Step 1

Step 2



Step 3

Answer sheet

Molarity of HCl solution: _____M

	Blank Titration (X)			Back titration (Y)		
Burette Reading (mL)	I	II	III	I	II	III
Initial						
Final						
Differance						

Constant Reading (Blank –X reading): _____mL (Back-Y reading): _____mL

Post lab Questions

1. Write the balanced chemical equation for the reaction between aspirin and NaOH once again (You have done it in pre-lab). Also write the balanced chemical equation for reaction between NaOH and HCl. Based on the balanced equation indicate the equivalence between the HCl, NaOH and aspirin

2. In the current experiment, the solution is heated after addition of NaOH. In your opinion, what the solution needs to be heated?

3. Please read the procedure for titration I and II and write the answers for following questions

- Volume of NaOH taken to prepare solution (under titration I) _____
- Volume of NaOH added to aspirin solution _____
- Total volume of diluted solution for Titration I: _____
- Total volume of diluted solution for Titration II: _____
- Volume of solution titrated for each reading in titration I: _____
- Volume of solution titrated for each reading in titration II _____
- Titrant used in titration I and II: _____ and _____respectively

Based on your above answers,

a) Explain whether you require or do not require molarity of NaOH to calculate the amount of aspirin.

b) Calculate the mmols of NaOH consumed by aspirin.

c) Calculate the amount of aspirin present (in g) in the tablet and it's percentage.

d) While doing the experiment, a pair of students heated the flask for 2 minutes and took the flask out for taking reading by mistake. While taking the next reading, they heated the flask for 5 minutes as given in the procedure. They observed that these two readings were significant different from one another. They realized what must have happened and thus for next reading they heated the flask for 8 mins and they observed that there is no difference between readings for flask 2 and 3.

i) In your opinion, what must be reason that the titration reading for flask 1 and 2 differed? State whether the reading obtained for flask 2 will be greater than or lower that reading obtained for first flask?

ii) Why did they decide to heat the flask for 8 mins instead of 5 minutes as given in the procedure?

e) Based on information filled in Question no. 3.

When do you perform back and blank titrations for a given experiment – list important aspects that you need to ensure before performing these titration methods.