Lesson 6: Back Titration

*Read through the lesson notes. You can write them out, print them or save them.

*Once you have tried to understand the lesson answer the questions that follow and self-evaluate your work by checking the answers.

Learning Intention

-Learn why back titration is carried out in a laboratory.

-Practice calculations back titration.

Background

We have now met three main types of titration; acid-base, redox and complexometric titrations. A back titration (sometimes referred to as indirect titration) is normally based on acid-base reactions. However, it is carried out when one of the substances of interest is insoluble.

Back Titration

Back titrations are used to find the number of moles of a substance by reacting it with an excess volume of a reactant of known concentration. The resulting mixture is then titrated to work out the number of moles of the reactant in excess. From the initial number of moles of that reactant, the number of moles used in the reaction can be determined. The initial number of moles of the substance being analysed can then be calculated. Two common examples that we will look at are:

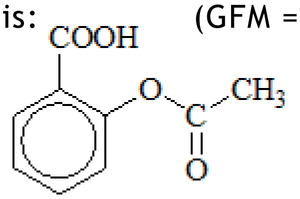
1. Determination of acetylsalicylic acid in aspirin tablets.

2. Determination of purity of marble.

1. Determination of acetylsalicylic acid in aspirin tablets

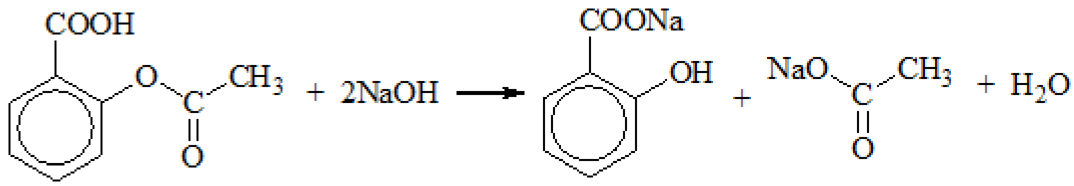
Since it is insoluble in water, aspirin has to be determined by a back titration technique. This involves treating a sample of accurately known mass or a specific number of tablets with a definite amount of sodium hydroxide, i.e. the volume and concentration of the alkali must be accurately known.

The structure of aspirin (acetylsalicylic acid) is: (GFM = 180g)



Worked Example

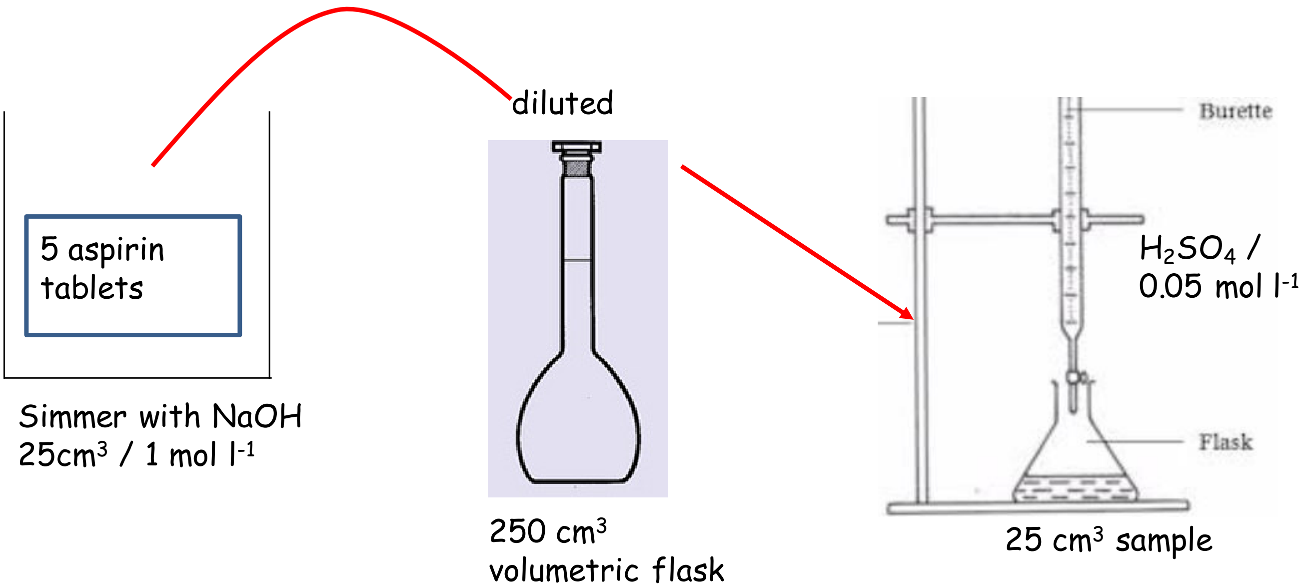
Five tablets were crushed and added to $25cm^3$ of 1 mol l^{-1} sodium hydroxide solution. The mixture was allowed to simmer for 30 minutes. This hydrolyses the acetylsalicylic acid.



The resulting solution was cooled and transferred to a $250cm^3$ volumetric flask and made up to the mark with distilled water. $25cm^3$ of this sample was titrated with 0.05 mol l^{-1} sulfuric acid solution.

$$2NaOH + H_2SO_4 \rightarrow Na_2SO_4 + 2H_2O$$

By titrating against a known concentration of sulfuric acid, this allows us to work back to calculate the number of moles of sodium hydroxide remaining after hydrolysing the aspirin tablet. As the original number of moles of sodium hydroxide is known, this finally allows us to calculate the mass of acetylsalicylic acid in the tablet.



The results of the titration are shown below.

	Rough titration	1st titration	2nd titration
Initial burette reading/cm^3	0·0	9·0	17·7
Final burette reading/cm^3	9·0	17·7	26·3
Volume used/cm^3	9·0	8·7	8·6

Step 1: moles of sulfuric acid.

n = c x v
n = 0.05 x 0.00865 ← **average titre volume from titration table**
n = <u>0.0004325 moles</u> of sulfuric acid

Step 2: moles of excess sodium hydroxide in volumetric flask.

2NaOH : H_2SO_4
x : 0.0004325
x = <u>8.65 x 10^{-4} moles</u> of NaOH reacted <u>in 25cm^3 sample</u>

Therefore: <u>8.65 x 10^{-3} moles</u> of excess NaOH in <u>250cm^3 standard flask</u>.

Step 3: moles of sodium hydroxide reacted with acetylsalicylic acid in the tablets.

n = c x v
n = 1 x 0.025
n = <u>0.025 moles</u> NaOH in Standard flask <u>at the beginning</u>

Therefore: 0.025 moles – 8.65 x 10^{-3} moles
= <u>0.01635 moles</u> NaOH reacted with acetylsalicylic acid

Step 4: Calculate the mass of acetylsalicylic acid in <u>one</u> aspirin tablet.

2NaOH : Acetylsalicylic acid
0.01635 : y
y = <u>0.008175 moles</u> acetylsalicylic acid reacted with NaOH

Mass of acetylsalicylic acid = n x GFM
= 0.008175 x 180
= 1.4715g ← **Mass in 5 tablets**

Therefore: mass of acetylsalicylic acid in <u>one</u> tablet is: <u>0.29g</u>

2. <u>Determination of purity of marble.</u>

Marble (calcium carbonate) is insoluble in water and so the calcium carbonate content has to be determined by a back titration technique. This involves treating a sample of marble of accurately known mass (1.62g) with a known quantity of hydrochloric acid, i.e. the volume and concentration of the acid must be known accurately. An excess of acid is used and the amount remaining after neutralising the calcium carbonate is determined by titrating it against a standard solution of sodium hydroxide.

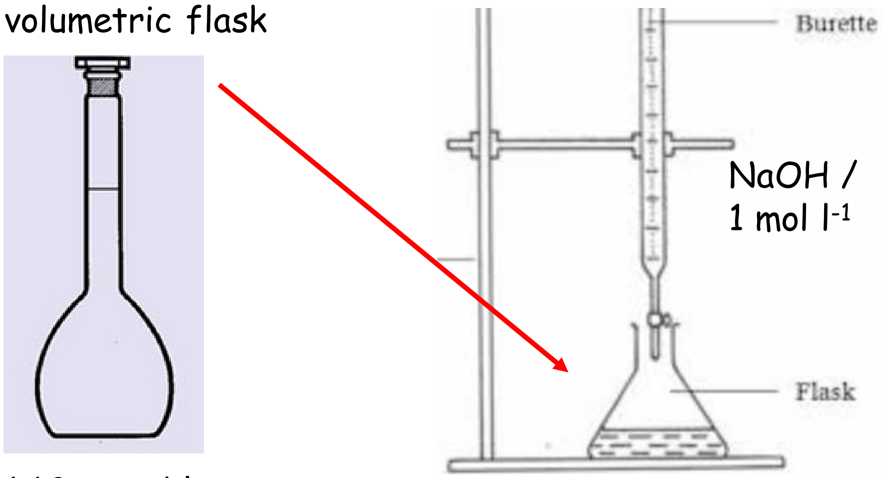
Reaction 1

$CaCO_3 + 2HCl \rightarrow CaCl_2 + H_2O + CO_2$

Reaction 2

$HCl + NaOH \rightarrow NaCl + H_2O$

250 cm^3
volumetric flask



1.62g marble
250cm^3 HCl 1 mol l^{-1}

25 cm^3 sample

The results of the titration are shown below.

	Rough titration	First Titration	Second titration
Initial burette reading/cm^3	0	22.1	0
Final burette reading/cm^3	22.1	43.9	21.9
Total volume NaOH added/cm^3	22.1	21.8	21.9

Step 1: moles of NaOH (from titration results)

n = c x v
n = 1 x 0.02185 (average of concordant titres)
n = 0.02185 moles

NaOH : HCl
1 mole : 1 mole
0.02185 moles : 0.02185 moles (25cm^3 sample)
X 10 = 0.2185 moles 250cm^3 in volumetric flask
This is the number of moles of HCl unreacted.

Step 2: moles of HCl at start

Number of moles of HCl at start = c x v
Number of moles of HCl at start = 1 x 0.25 (total volume of volumetric flask)
Number of moles of HCl at start = 0.25 moles

Step 3: moles of HCl reacted with marble

Number of moles of HCl at start – number of moles of HCl unreacted
0.25 moles – 0.2185 moles
0.0315 moles (that have reacted with marble)

Step 4: Mass and purity of marble

2HCl : $CaCO_3$
2 moles : 1 mole
0.0315 moles : 0.01575 moles

Mass of $CaCO_3$ in marble = n x GFM
Mass of $CaCO_3$ in marble = 0.01575 x 100.1 = 1.58g

$\% = \frac{1.58}{1.62} \times 100 = 97.3\%$

This indicates that the sample of marble has 2.7% impurities.

→Watch the following clips.

https://drive.google.com/file/d/1v6IkHwy368WdDtAIS_B90r4zJ7cUC7G9/view?usp=sharing

https://www.youtube.com/watch?v=oHVSCrZ3Aj4

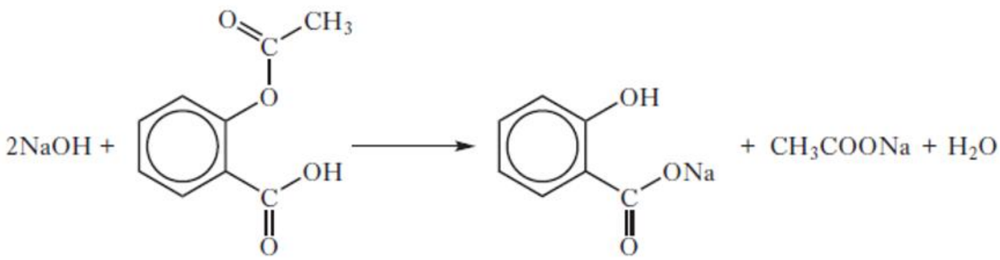
→ Read Scholar Heriot-Watt/ Researching Chemistry Section 5.5.

→ Read Bright Red text book page 84 and 85.

→ Answer the questions from Sheet 4.6 and check the answers when you have completed them.

4.6 Back Titration

1. The active ingredient in aspirin tablets is acetylsalicylic acid, $C_9H_8O_4$. The acetylsalicylic acid content of an aspirin tablet can be determined using a back titration.
Four aspirin tablets were crushed and added to 25.0 cm^3 of 1.00 mol l^{-1} sodium hydroxide solution. The mixture was heated and allowed to simmer for 30 minutes.



The resulting mixture was allowed to cool before being transferred to a 250 cm^3 standard flask and made up to the mark with deionised water.
25.0 cm^3 samples of this solution were titrated with 0.050 mol l^{-1} sulfuric acid.

$$2NaOH + H_2SO_4 \rightarrow Na_2SO_4 + 2H_2O$$

The results of the titration are shown in the table below.

	1st attempt	2nd attempt	3rd attempt
Initial burette reading (cm^3)	0.0	13.2	26.0
Final burette reading (cm^3)	13.2	26.0	38.9
Titre (cm^3)	13.2	12.8	12.9

a) Calculate the number of moles of sulfuric acid in the average titre.

b) Calculate the number of moles of excess sodium hydroxide in the volumetric flask.

c) Calculate the number of moles of sodium hydroxide which reacted with the acetylsalicylic acid.

d) Calculate the number of moles of acetylsalicylic acid which reacted with the sodium hydroxide.

e) Calculate the mass of acetylsalicylic acid in **one** aspirin tablet.
(GFM acetylsalicylic acid = 180g)

2. In an experiment 0.25 g of an impure sample of magnesium carbonate was added to 40 cm^3 of 0.16 mol l^{-1} hydrochloric acid. 8.1 cm^3 of 0.11 mol l^{-1} sodium hydroxide solution was required to neutralise the excess hydrochloric acid. The reactions involved were:

$$MgCO_3 + 2HCl \rightarrow MgCl_2 + H_2O + CO_2$$

$$NaOH + HCl \rightarrow NaCl + H_2O$$

a) Show by calculation that 0.0055 moles of hydrochloric acid reacted with the magnesium carbonate.

b) Calculate the percentage purity of the magnesium carbonate.

3. To analyse the calcium carbonate content in indigestion tablets, a student carried out the following back titration.

Five indigestion tablets containing calcium carbonate were crushed up and an excess of 100 cm^3 1.2 mol l^{-1} hydrochloric acid was added. This mixture was gently heated to allow it to react.

$$CaCO_3 + 2HCl \rightarrow CaCl_2 + H_2O + CO_2$$

The next step was to titrate the mixture with 0.8 mol l^{-1} NaOH. It was found that the average titre of NaOH was 32.6 cm^3.

$$NaOH + HCl \rightarrow NaCl + H_2O$$

Use the student's results to calculate the mass of calcium carbonate in **one** indigestion tablet.

4. The lead ore cerussite consists mainly of lead (II) carbonate. 2.00 g of crushed ore was added to 25 cm^3 1.0 mol l^{-1} of nitric acid in a beaker. When no more reaction was observed the mixture was filtered into a 250 cm^3 volumetric flask and made up to the mark with deionised water.
Then 25 cm^3 portions of this solution were titrated with standardised 0.1 mol l^{-1} sodium hydroxide. The average titre to reach the end point was 12.7 cm^3.

$$PbCO_3 + 2HNO_3 \rightarrow Pb(NO_3)_2 + H_2O + CO_2$$

$$NaOH + HNO_3 \rightarrow NaNO_3 + H_2O$$

Calculate the percentage by mass of lead carbonate present in the ore.

5. 1.58g of crushed eggshell was placed in a beaker containing 50.0 cm^3 of 1.00 mol l^{-1} hydrochloric acid.

$$CaCO_3(s) + 2HCl\,(aq) \rightarrow CaCl_2(aq) + CO_2(g)\;\; H_2O(\ell)$$

Once the reaction was complete, the solution was quantitatively transferred to a 250cm^3 standard flask and made up to the mark with distilled water.

25.0 cm^3 samples of the solution were titrated with 0.1 mol l^{-1} sodium hydroxide solution. The average titre was 17.85 cm^3.

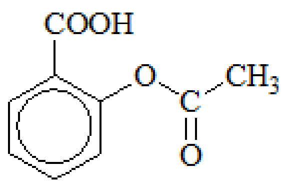
$$NaOH(aq) + HCl\,(aq) \rightarrow NaCl(aq) + H_2O(\ell)$$

(a) Calculate the number of moles of hydrochloric acid left unreacted in the standard flask.

(b) Using the results, calculate the percentage, by mass, of calcium carbonate present in the eggshell.

(c) The procedure gave a mass of calcium carbonate that was greater than expected.
Suggest a reason for this and describe an improvement that should be made to the procedure to give a mass closer to the expected value.

6. For an Advanced Higher Chemistry project, a pupil was asked to investigate the mass of acetylsalicylic acid in different brands of aspirin tablets. The pupil made the assumption that the aspirin tablets could be titrated directly with laboratory sodium hydroxide solution. The structure of acetylsalicylic acid is shown below.



a) By considering the structure of acetylsalicylic acid, why does it initially seem reasonable to titrate the different brands of aspirin directly with laboratory sodium hydroxide solution?

b) In practice, the pupil did not titrate the aspirin tablets directly with laboratory sodium hydroxide solution. Explain why they had to carry out a back titration for this investigation.

c) Explain in general terms how the analytical procedure of back titration is carried out.