CHEMICAL ANALYSIS

Titration
Titration is a procedure for determining the concentration of a solution by allowing a carefully measured volume to react with a standard (of known concentration) solution of another substance. An acid-base titration is quite common, and is based on the neutralization reaction between an acid and a base. In the laboratory, such a titration can be monitored using an acid-base indicator or an instrument called a pH meter.

(1) A known volume of acid is pipetted into a conical flask and universal indicator added. The acid is titrated with the alkali in the burette

(2) until the indicator turns green.

(3). The volume of alkali needed for neutralisation is then noted, this is called the endpoint. (1-3) are repeated with both known volumes mixed together BUT without the contaminating indicator.

![Diagram depicting titration process]

The volume of standard solution required for complete reaction can be used to calculate the concentration of the unknown solution. Let's consider a titration of HCl with KOH. Results of a titration indicate that 29.7 mL of a 0.100M solution of KOH was required to completely neutralize a 15.0 cm³ sample of HCl. We begin by writing a balanced equation for the titration reaction.

\[
\text{HCl(aq) + KOH(aq)} \rightarrow \text{H}_2\text{O(l) + KCl(aq)}
\]

Because we know the concentration and the volume of the KOH solution, we can calculate the number of moles of KOH used in the reaction.

\[
\text{Number of moles of KOH in 29.7 cm}^3 = \frac{29.7 \times 0.1}{1000} = 2.97 \times 10^{-3} \text{ mol KOH}
\]

The number of moles of KOH is related to the number of moles of HCl by the stoichiometric coefficients of the balanced chemical equation. In this case, there is a 1:1 ratio. Don't be tempted to omit this step, however, because the ratio is not always 1:1.

\[
2.97 \times 10^{-3} \text{ mol KOH (1 mol HCl / 1 mol KOH)} = 2.97 \times 10^{-3} \text{ mol HCl}
\]
We can now calculate the concentration of the HCl solution by dividing moles by volume.

\[
2.1 \times 10^{-3} \text{ mol HCl} / 0.015 \text{ dm}^3 = 0.198 \text{ mol/dm}^3 \text{ HCl} = 0.198 \text{ M HCl}
\]

**Titration of sulphuric acid with sodium hydroxide.**

**Problem No. 1**

30 cm\(^3\) of 0.1 mol/dm\(^3\) NaOH (aq) reacted completely with 25 cm\(^3\) of H\(_2\)SO\(_4\) (aq) in a titration flask. Calculate the concentration of H\(_2\)SO\(_4\) in (a) mol/dm\(^3\) and (b) in g/dm\(^3\). The equation for the reaction is

**DATA**

- Concentration Of NaOH = 0.1 mol/dm\(^3\)
- Concentration of H\(_2\)SO\(_4\) = unknown
- Volume of NaOH = 30 cm\(^3\)
- Volume of H\(_2\)SO\(_4\) = 25 cm\(^3\)

**Step 1:** First find the number of moles of NaOH use in titration

1 dm\(^3\) contain 0.1 mol

0.03 dm\(^3\) (30 cm\(^3\)) will contain \(0.03 \times 0.1 = 0.003\) moles (in 30 ml)

**Step 2:** Write the chemical equation for the reaction.

\[2\text{NaOH(aq)} + \text{H}_2\text{SO}_4(aq) \rightarrow \text{Na}_2\text{SO}_4(aq) + 2\text{H}_2\text{O(l)}\]

**Step 3:** From the equation find the ratio of number of moles of H\(_2\)SO\(_4\) to the number of moles of NaOH

\[
\text{NaOH} : \text{H}_2\text{SO}_4 = 2 : 1
\]

**Step 4:** Use the ratio to find the number of moles of H\(_2\)SO\(_4\) that reacted.

\[
\frac{0.003}{2} : 0.0015
\]

**Step 5:** Find the concentration of H\(_2\)SO\(_4\) in moles/dm\(^3\).

0.0015 moles are present in 0.025 dm\(^3\)

\[
\frac{0.0015 \times 1}{0.025} = 0.06 \text{ moles/dm}^3
\]

**Step 6:** Find the concentration of H\(_2\)SO\(_4\) in g/dm\(^3\)
0.06 x 98 = 5.88 g/dm³

**Titration of iron (II) sulphate with potassium manganate (VII).**

25.0 cm³ of FeSO₄(aq), acidified with sulphuric acid, required 27.5 cm³ of 0.0200 mol/dm³ KMnO₄ (aq) for reaction in a titration. Calculate the concentration of FeSO₄ (aq).

**Step 1:** Find the number of moles of KMnO₄ used in titration

0.02 moles are present in 1 dm³
x moles are present in 0.0275 dm³ (27.5 cm³)

\[
\frac{0.02 \times 0.0275}{1} = 0.00055 \text{ molers in } 27.5 \text{ cm}³
\]

**Step 2:** Write the chemical equation for the reaction.

\[
2\text{KMnO}_4 (aq) + 10\text{FeSO}_4 (aq) + 8 \text{H}_2\text{SO}_4 (aq) \rightarrow \text{K}_2\text{SO}_4 (aq) + 2\text{MnSO}_4 (aq) + 5\text{Fe}_2(\text{SO}_4)_3 (aq) + 8\text{H}_2\text{O} (l)
\]

**Step 3:** From the equation find the ratio of number of moles of FeSO₄ to the number of moles of KMnO₄.

\[
\text{KMnO}_4 : \text{FeSO}_4 = 1 : 5
\]

**Step 4:** Use the ratio to find the number of moles of FeSO₄ that reacted in the titration.

\[
\frac{0.00055}{0.0275} = 0.11 \text{ moles/dm}³
\]

**Step 5:** Find the concentration of FeSO₄ (aq) in mol/dm³.

0.025 dm³ contains 0.00275 moles
1 dm³ contains

\[
\frac{0.00275 \times 0.025}{1} = 0.11 \text{ moles/dm}³
\]
USES OF TITRATION IN ANALYSIS

1. Identification of Acids and Alkalis.

An acid has the formula $H_2XO_4$. One mole of $H_2XO_4$ reacts with two moles of NaOH. A solution of the acid contains 5.0 g/ dm$^3$ of $H_2XO_4$. In a titration, 25.0 cm$^3$ of the acid reacted with 25.5 cm$^3$ of 0.1 mol/ dm$^3$ NaOH (aq). Calculate the concentration of the acid in mol/ dm$^3$ and hence calculate the relative molecular mass of the acid.

Solution:

Concentration Of NaOH $= 0.1$ mol/dm$^3$
Concentration of $H_2XO_4 = 50$ g/dm$^3$
Volume of NaOH $= 25$ cm$^3$
Volume of $H_2SO_4 = 25$ cm$^3$

Step 1: Write the balanced equation

$$H_2XO_4 + 2NaOH \rightarrow Na_2XO_4 + 2H_2O$$

Step 2 : Find the numbers of moles of NaOH used in titration

No. of moles of NaOH used in titration $= concentration \times vol. \ in \ dm^3$

$$= 0.1 \times \frac{25.5}{1000}$$

No. of moles of $H_2XO_4 = \frac{1}{2} \times$ no. of moles of NaOH

$$= \frac{1}{2} \times 0.1 \times 25.5 \times \frac{1000}{1000}$$

Concentration of the acid $= \frac{no. \ of \ moles \ of \ H_2XO_4}{Vol. \ of \ acid \ in \ dm^3}$

$$= \frac{1}{2} \times 0.1 \times 25.5 \times \frac{1000}{1000}$$

$$= \frac{25.0}{1000}$$

$$= 0.051 \ \text{mol/dm}^3$$

1 dm$^3$ acid solution contains 0.051 mol and 5.0 g of $H_2XO_4$.
So 0.051 mol of $H_2XO_4$ has a mass of 5.0 g of $H_2XO_4$.
And 1 mol. Of $H_2XO_4$ has a mass of $5.0/0.051 = 98$ g.
Hence the relative molecular mass of $\text{H}_2\text{ XO}_4$ is 98.

The relative atomic mass of $\text{X} = 98 - 66 = 32$.
So $\text{X}$ is sulphur and the acid is $\text{H}_2\text{SO}_4$.

2. **Percentage Purity of Compounds**

Solution of $\text{X}$ contains 5.00 g of impure sulphuric acid dissolved in 1 dm$^3$ of solution. 25.0 cm$^3$ of solution $\text{X}$ required 23.5 cm$^3$ of 0.100 mol/dm$^3$ NaOH for the reaction in titration. Calculate the percentage purity of acid.

\[
\text{Percentage purity} = \frac{\text{mass of actual acid} / \text{dm}^3}{\text{Mass of impure acid}/ \text{dm}^3} \times 100
\]

Solution:

No. of moles of NaOH used in titration = \(\frac{23.5 \times 0.100}{1000}\) mol.

The equation is

$$\text{H}_2\text{SO}_4 \ + 2\text{NaOH} \longrightarrow \text{Na}_2\text{SO}_4 \ + 2\text{H}_2\text{O}$$

From the equation,

No. of moles of $\text{H}_2\text{SO}_4$ = \(\frac{1}{2} \times \) no. of moles of NaOH

= \(\frac{1}{2} \times \frac{23.5}{1000} \times 0.100\) mol.

So the concentration of $\text{H}_2\text{SO}_4$

= \(\frac{\text{no of moles}}{\text{vol. in dm}^3}\)

= \(\frac{1}{2} \times \frac{23.5}{1000} \times 0.100\)

= \(\frac{25.5}{1000}\)

= 0.047 mol/dm$^3$.

Hence the no. of grams of $\text{H}_2\text{SO}_4$ in 1 dm$^3$

= 0.047 \times \text{Mr of } \text{H}_2\text{SO}_4

= 0.047 \times 98

= 4.61 g.

Hence the percentage purity = \(\frac{4.61}{5.00} \times 100 = 92.2\%\)
3. **Formulas of compounds**

Solution Y contains 30.0 g of FeSO$_4$. $x$H$_2$O. In a titration 25.0 cm$^3$ of solution Y (FeSO$_4$. $x$H$_2$O) reacted with 27.0 cm$^3$ of 0.02 mol/dm$^3$ KMnO$_4$. In the reaction 5 moles of Y reacts with one mole of KMnO$_4$. Calculate the concentration of Y in mol/dm$^3$ and hence find the value of $x$.

No. of moles of KMnO$_4$ used in the titration = 27.0 x 0.020 mol = 0.00054 mol

No. of moles of FeSO$_4$. $x$H$_2$O that reacted with KMnO$_4$ in titration.

Conc. of FeSO$_4$. xH$_2$O = 0.0027 x 1000

= 0.108 mol/dm$^3$

Hence 0.108 mol of FeSO$_4$.xH$_2$O = 30 g.
Therefore 1 mole of FeSO$_4$. xH$_2$O will be equal to 30/ 0.108 = 278 g.

Mr. Of FeSO$_4$ = 152
Mr. Of FeSO$_4$.xH$_2$O = 278
Therefore $x$.H$_2$O = 278- 152 = 126
Mr. of H$_2$O = 18
Mr. of xH$_2$O = 126
Therefore $x$ = 126 / 18 = 7

Therefore formula will be FeSO$_4$.7H$_2$O

4. **Numbers of Reacting Moles in an equation.**

xH$_2$O$_2$ + yKMnO$_4$ + acid --------------> Product

In a titration, 25.0 cm$^3$ of 0.0400mol/dm$^3$ H$_2$O$_2$ reacted with 20.0 cm$^3$ of 0.0200 mol/dm$^3$ KMnO$_4$. Find the value of $x$ and $y$ in the outline equation above.

No. of moles of H$_2$O$_2$ used in the titration = 25.0 x 0.0400 mol = 0.001 mol.

No. of moles of KMnO$_4$ used in the titration = 20.0 x 0.0200 mol = 0.0004 mol.
So we can say that 0.001 moles of $\text{H}_2\text{O}_2$ reacts with 0.004 moles of $\text{KMnO}_4$.

So 1 mole of $\text{KMnO}_4$ would react with $\frac{0.001}{0.004} = 2.5$ mole

Hence the ratio of $x$ to $y$ is $1:2.5 = 2:5$.

So $x = 2$ and $y = 5$.

**Precipitation Reactions and Solubility Rules**

To predict whether a precipitation reaction will occur upon mixing aqueous solutions, you must know the solubility of each of the potential products. A substance that has a low solubility in water will likely form a precipitate in aqueous solution. A substance with a high solubility in water will not precipitate in solution.

The following solubility guidelines will be helpful in predicting precipitates:

1. A compound containing one of the following cations is probably soluble:
   - Group 1A cation: $\text{Li}^+$, $\text{Na}^+$, $\text{K}^+$, $\text{Rb}^+$, $\text{Cs}^+$
   - Ammonium ion: $\text{NH}_4^+$

2. A compound that contains one of the following anions is probably soluble:
   - Halide: $\text{Cl}^-$, $\text{Br}^-$, $\text{I}^-$
   - Except $\text{Ag}^+$, $\text{Hg}^{2+}$, $\text{Pb}^{2+}$ compounds
   - Nitrate ($\text{NO}_3^-$), perchlorate ($\text{ClO}_4^-$), acetate ($\text{CH}_3\text{COO}^-$), sulfate ($\text{SO}_4^{2-}$)
   - Except $\text{Ba}^{2+}$, $\text{Hg}^{2+}$, $\text{Pb}^{2+}$ sulfates

3. Most compounds that contain the following anions are insoluble unless they contain a Group 1A cation, ammonium ion:
   - Hydroxide ($\text{OH}^-$), oxide ($\text{O}^2$), carbonate ($\text{CO}_3^{2-}$), phosphate ($\text{PO}_4^{3-}$), chromate($\text{CrO}_4^{2-}$), sulfide($\text{S}^2$)

To predict the outcome when combining aqueous solutions of ionic compounds:

1. Write the complete molecular equation.
2. Determine whether the products will be soluble or insoluble by consulting the solubility guidelines.
3. Write the complete ionic equation, separating the soluble products into their component ions.
4. Cancel and remove the spectator ions. The resulting net ionic equation must show the formation of an insoluble solid product in a precipitation reaction.