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## 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.

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 0.100 M solution of KOH was required to completely
neutralize a $15.0 \mathrm{~cm}^{3}$ sample of HCl . We begin by writing a balanced equation for the titration reaction.

$$
\begin{array}{cl}
\mathrm{HCl}(\mathrm{aq})+\mathrm{KOH}(\mathrm{aq})-\cdots--\cdots----\rightarrow \mathrm{H}_{2} \mathrm{O}(\mathrm{l})+\mathrm{KCl}(\mathrm{aq}) \\
15.0 \mathrm{~mL} & 29.7 \mathrm{~cm}^{3} \\
\mathrm{M} ? & 0.100 \mathrm{M}
\end{array}
$$

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.

$$
\begin{aligned}
\text { Number of moles of } \mathrm{KOH} \text { in } 29.7 \mathrm{~cm}^{3} & =\frac{29.7 \times 0.1}{1000} \\
& =2.97 \times 10^{-3} \mathrm{~mol} \mathrm{KOH}
\end{aligned}
$$

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.
$2.97 \times 10^{-3} \mathrm{~mol} \mathrm{KOH}(1 \mathrm{~mol} \mathrm{HCl} / 1 \mathrm{~mol} \mathrm{KOH})=2.97 \times 10^{-3} \mathrm{~mol} \mathrm{HCl}$

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We can now calculate the concentration of the HCl solution by dividing moles by volume.

$$
2.1 \times 10^{-3} \mathrm{~mol} \mathrm{HCl} / 0.015 \mathrm{dm}^{3}=0.198 \mathrm{~mol} / \mathrm{dm}^{3} \mathrm{HCl}=0.198 \mathrm{M} \mathrm{HCl}
$$

## Titration of sulphuric acid with sodium hydroxide.

## Problem No. 1

$30 \mathrm{~cm}^{3}$ of $0.1 \mathrm{~mol} / \mathrm{dm}^{3} \mathrm{NaOH}$ (aq) reacted completely with $25 \mathrm{~cm}^{3}$ of $\mathrm{H}_{2} \mathrm{SO}_{4}$ (aq) in a titration flask. Calculate the concentration of $\mathrm{H}_{2} \mathrm{SO}_{4}$ in (a) $\mathrm{mol} / \mathrm{dm}^{3}$ and (b) in $\mathrm{g} / \mathrm{dm}^{3}$. The equation for the reaction is

## DATA

Concentration Of $\mathrm{NaOH}=0.1 \mathrm{~mol} / \mathrm{dm}^{3}$
Concentration of $\mathrm{H}_{2} \mathrm{SO}_{4}=$ unknown
Volume of $\mathrm{NaOH}=30 \mathrm{~cm}^{3}$
Volume of $\mathrm{H}_{2} \mathrm{SO}_{4}=25 \mathrm{~cm}^{3}$
Step 1: First find the number of moles of NaOH use in titration

$$
\begin{aligned}
& 1 \mathrm{dm} 3 \text { contain } 0.1 \mathrm{~mol} \\
& 0.03 \mathrm{dm} 3(30 \mathrm{~cm} 3) \text { will contain } 0.03 \times 0.1=0.003 \text { moles }(\text { in } 30 \mathrm{ml})
\end{aligned}
$$

Step 2:Write the chemical equation for the reaction.

$$
2 \mathrm{NaOH}(\mathrm{aq})+\mathrm{H}_{2} \mathrm{SO}_{4}(\mathrm{aq})---\cdots-\cdots-\cdots-\cdots \mathrm{Na}_{2} \mathrm{SO}_{4}(\mathrm{aq})+2 \mathrm{H}_{2} \mathrm{O}(\mathrm{l})
$$

Step 3: From the equation find the ratio of number of moles of $\mathrm{H}_{2} \mathrm{SO}_{4}$ to the number of moles of NaOH

$$
\begin{array}{llc}
\mathrm{NaOH} & : & \mathrm{H}_{2} \mathrm{SO}_{4} \\
2 & : & 1
\end{array}
$$

Step 4: Use the ration to find the number of moles of $\mathrm{H}_{2} \mathrm{SO}_{4}$ that reacted.

| NaOH | $:$ | $\mathrm{H}_{2} \mathrm{SO}_{4}$ |
| :--- | :--- | :--- |
| $0.003:$ | 0.0015 |  |

Step 5: Find the concentration of $\mathrm{H}_{2} \mathrm{SO}_{4}$ in moles/ $\mathrm{dm}^{3 .}$
0.0015 moles are present in $0.025 \mathrm{dm}^{3}$
x moles are present in $1 \mathrm{dm}^{3}$

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

Step 6: Find the concentration of $\mathrm{H}_{2} \mathrm{SO}_{4}$ in $\mathrm{g} / \mathrm{dm}^{3}$
$0.06 \times 98=5.88 \mathrm{~g} / \mathrm{dm}^{3}$

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

$25.0 \mathrm{~cm}^{3}$ of $\mathrm{FeSO} 4(\mathrm{aq})$, acidified with sulphuric acid, required $27.5 \mathrm{~cm}^{3}$ of $0.0200 \mathrm{~mol} / \mathrm{dm}^{3} \mathrm{KMnO}_{4}(\mathrm{aq})$ for reaction in a titration. Calculate the concentration of $\mathrm{FeSO}_{4}(\mathrm{aq})$.

Step1: Find the number of moles of $\mathrm{KMnO}_{4}$ used in titration
0.02 moles are present in 1 dm 3
$x$ moles are present in $0.0275 \mathrm{dm} 3\left(27.5 \mathrm{~cm}_{3}\right)$

$$
\frac{0.02 \times 0.0275}{1}=0.00055 \text { molers in } 27.5 \mathrm{~cm} 3
$$

Step 2: Write the chemical equation for the reaction.
$2 \mathrm{KMnO}_{4}(\mathrm{aq})+10 \mathrm{FeSO}_{4}(\mathrm{aq})+8 \mathrm{H}_{2} \mathrm{SO}_{4}(\mathrm{aq})-\cdots-\cdots----\mathrm{K}_{2} \mathrm{SO}_{4}(\mathrm{aq})+2 \mathrm{MnSO}_{4}(\mathrm{aq})+$

Step 3: From the equation find the ratio of number of moles of $\mathrm{FeSO}_{4}$ to the number of moles of $\mathrm{KMnO}_{4}$.

| $\mathrm{KMnO}_{4}$ | $:$ | $\mathrm{FeSO}_{4}$ |
| :---: | :---: | :---: |
| 1 | $:$ | 5 |

Step 4: Use the ratio to find the number of moles of $\mathrm{FeSO}_{4}$ that reacted in the titration.

| $\mathrm{KMnO}_{4}$ | $:$ | $\mathrm{FeSO}_{4}$ |
| :--- | :--- | :--- |
| 0.00055 | $:$ | 00275 |

Step 5: Find the concentration of $\mathrm{FeSO}_{4}(\mathrm{aq})$ in $\mathrm{mol} / \mathrm{dm}^{3}$.
$0.025 \mathrm{dm}^{3}$ contains 0.00275 moles
$1 \mathrm{dm}^{3}$ contains

$$
\frac{0.00275 \times 0.025}{1}=0.11 \mathrm{moles} / \mathrm{dm}^{3}
$$

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## USES OF TITRATION IN ANALYSIS

## 1. Identification of Acids and Alkalis.

An acid has the formula $\mathrm{H}_{2} \mathrm{XO}_{4}$. One mole of $\mathrm{H}_{2} \mathrm{XO}_{4}$ reacts with two moles of NaOH . A solution of the acid contains $5.0 \mathrm{~g} / \mathrm{dm}^{3}$ of $\mathrm{H}_{2} \mathrm{XO}_{4}$. In a titration, $25.0 \mathrm{~cm}^{3}$ of the acid reacted with $25.5 \mathrm{~cm}^{3}$ of $0.1 \mathrm{~mol} /$ $\mathrm{dm}^{3} \mathrm{NaOH}(\mathrm{aq})$. Calculate the concentration of the acid in $\mathrm{mol} / \mathrm{dm}^{3}$ and hence calculate the relative molecular mass of the acid.

Solution:
Concentration Of $\mathrm{NaOH}=0.1 \mathrm{~mol} / \mathrm{dm}^{3}$
Concentration of $\mathrm{H}_{2} \mathrm{XO}_{4}=50 \mathrm{~g} / \mathrm{dm}^{3}$
Volume of $\mathrm{NaOH}=25 \mathrm{~cm}^{3}$
Volume of $\mathrm{H}_{2} \mathrm{SO}_{4} \quad=25 \mathrm{~cm}^{3}$

Step 1: Write the balanced equation

$$
\mathrm{H}_{2} \mathrm{XO}_{4}+2 \mathrm{NaOH}--------------\mathrm{Na}_{2} \mathrm{XO}_{4}+2 \mathrm{H}_{2} \mathrm{O}
$$

Step 2 : Find the numbers of moles of NaOH used in titration
No. of moles of NaOH used in titration $\quad=$ concentration x vol. in $\mathrm{dm}^{3}$

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

No. of moles of $\mathrm{H}_{2} \mathrm{XO}_{4}$

Concentration of the acid

$$
=\underline{\text { no. of moles of }} \mathrm{H}_{2} \mathrm{XO}_{4}
$$

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

$$
\underline{25.0}
$$

$$
1000
$$

$$
=0.051 \mathrm{~mol} / \mathrm{dm}^{3}
$$

$1 \mathrm{dm}^{3}$ acid solution contains 0.051 mol and 5.0 g of $\mathrm{H}_{2} \mathrm{XO}_{4}$.
So 0.051 mol of $\mathrm{H}_{2} \mathrm{XO}_{4}$ has a mass of 5.0 g of $\mathrm{H}_{2} \mathrm{XO}_{4}$.
And 1 mol . Of $\mathrm{H}_{2} \mathrm{XO}_{4}$ has a mass of $5 \cdot 0 / 0.051=98 \mathrm{~g}$.

Hence the relative molecular mass of $\mathrm{H}_{2} \mathrm{XO}_{4}$ is 98 .
The relative atomic mass of $\mathrm{X}=98-66=32$.
So X is sulphur and the acid is $\mathrm{H}_{2} \mathrm{SO}_{4}$.

## 2. Percentage Purity of Compounds

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

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

Solution:
No. of moles of NaOH used in titration $=\frac{23.5}{1000} \times 0.100 \mathrm{~mol}$.
The equation is

$$
\mathrm{H}_{2} \mathrm{SO}_{4}+2 \mathrm{NaOH}---\cdots-\cdots-\cdots-\cdots \mathrm{Na}_{2} \mathrm{SO}_{4}+2 \mathrm{H}_{2} \mathrm{O}
$$

From the equation,

$$
\text { No. of moles of } \begin{aligned}
\mathrm{H}_{2} \mathrm{SO}_{4} & =1 / 2 \times \text { no. of moles of } \mathrm{NaOH} \\
& =1 / 2 \times \frac{23.5}{1000} \times 0.100 \mathrm{~mol} .
\end{aligned}
$$

So the concentration of $\mathrm{H}_{2} \mathrm{SO}_{4}$

$$
\begin{aligned}
& =\frac{\text { no of moles }}{\text { vol. } \mathrm{in}^{3}} \\
& =1 / 2 \times \frac{23.5}{1000} \times 0.100 \\
& \frac{25.5}{1000} \\
& =0.047 \mathrm{~mol} / \mathrm{dm}^{3} .
\end{aligned}
$$

Hence the no. of grams of $\mathrm{H}_{2} \mathrm{SO}_{4}$ in $1 \mathrm{dm}^{3}$

$$
\begin{aligned}
& =0.047 \times \mathrm{Mr} \text { of } \mathrm{H}_{2} \mathrm{SO}_{4} \\
& =0.047 \times 98 \\
& =4.61 \mathrm{~g} .
\end{aligned}
$$

Hence the percentage purity $=\frac{4.61}{5.00} \times 100=92.2 \%$

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## 3. Formulas of compounds

Solution Y contains 30.0 g of $\mathrm{FeSO}_{4} \cdot \mathrm{xH}_{2} \mathrm{O}$. In a titration $25.0 \mathrm{~cm}^{3}$ of solution $\mathrm{Y}\left(\mathrm{FeSO}_{4} \cdot \mathrm{xH}_{2} \mathrm{O}\right)$ reacted with $27.0 \mathrm{~cm}^{3}$ of $0.02 \mathrm{~mol} / \mathrm{dm}^{3} \mathrm{KMnO}_{4}$. In the reaction 5 moles of Y reacts with one mole of $\mathrm{KMnO}_{4}$. Calculate the concentration of Y in $\mathrm{mol} / \mathrm{dm}^{3}$ and hence find the value of x .

No. of moles of $\mathrm{KMnO}_{4}$ used in the titration $\quad=\frac{27.0}{1000} \times 0.020 \mathrm{~mol}=0.00054 \mathrm{~mol}$

No. of moles of $\mathrm{FeSO}_{4} . \mathrm{xH}_{2} \mathrm{O}$ that reacted with $\mathrm{KMnO}_{4}$ in titration.

$$
\begin{aligned}
& =\frac{0.0027}{25} \times 1000 \\
& =0.108 \mathrm{~mol} / \mathrm{dm} 3
\end{aligned}
$$

Conc. of $\mathrm{FeSO}_{4} . \mathrm{xH}_{2} \mathrm{O}$

Hence 0.108 mol of $\mathrm{FeSO}_{4} \cdot \mathrm{xH}_{2} \mathrm{O}=30 \mathrm{~g}$.
Therefore 1 mole of $\mathrm{FeSO}_{4} \cdot \mathrm{xH}_{2} \mathrm{O}$ will be equal to $30 / 0.108=278 \mathrm{~g}$.
Mr . Of $\mathrm{FeSO}_{4}=152$
Mr. Of $\mathrm{FeSO}_{4} \cdot \mathrm{xH}_{2} \mathrm{O}=278$
Therefore $\mathrm{x} . \mathrm{H}_{2} \mathrm{O}=278-152=126$
Mr. of $\mathrm{H}_{2} \mathrm{O}=18$
Mr. of $\mathrm{xH}_{2} \mathrm{O}=126$
Therefore $\mathrm{x}=126 / 18=7$
Therefore formula will be $\mathrm{FeSO}_{4} .7 \mathrm{H}_{2} \mathrm{O}$

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

$\mathrm{xH}_{2} \mathrm{O}_{2}+\mathrm{yKMnO}_{4}+$ acid $---------------\rightarrow$ Product

In a titration, $25.0 \mathrm{~cm}^{3}$ of $0.0400 \mathrm{~mol} / \mathrm{dm}^{3} \mathrm{H}_{2} \mathrm{O}_{2}$ reacted with $20.0 \mathrm{~cm}^{3}$ of $0.0200 \mathrm{~mol} / \mathrm{dm}^{3} \mathrm{KMnO}_{4}$. Find the value of $x$ and $y$ in the outline equation above.

No. of moles of $\mathrm{H}_{2} \mathrm{O}_{2}$ used in the titration $=\frac{25.0}{1000} \times 0.0400 \mathrm{~mol}=0.001 \mathrm{~mol}$.

No. of moles of $\mathrm{KMnO}_{4}$ used in the titration $=\frac{20.0}{1000} \times 0.0200 \mathrm{~mol}=0.0004 \mathrm{~mol}$.

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So we can say that 0.001 moles of $\mathrm{H}_{2} \mathrm{O}_{2}$ reacts with 0.004 moles of $\mathrm{KMnO}_{4}$.
So 1 mole of $\mathrm{KMnO}_{4}$ would react with $\underline{0.001}=2.5$ mole
0.0004

Hence the ratio of $\mathrm{x}: \mathrm{y}$ is $1: 2.5=2: 5$.
So $\mathrm{x}=2$ and $\mathrm{y}=5$.

## 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: $\mathrm{Li}^{+}, \mathrm{Na}^{+}, \mathrm{K}^{+}, \mathrm{Rb}^{+}, \mathrm{Cs}^{+}$Ammonium ion: $\mathrm{NH}_{4}^{+}$
2. A compound that contains one of the following anions is probably soluble:

Halide: $\mathrm{Cl}^{-}, \mathrm{Br}^{-}, \mathrm{I}^{-}$
Except $\mathrm{Ag}^{+}, \mathrm{Hg}^{2+}, \mathrm{Pb}^{2+}$ compounds
Nitrate $\left(\mathrm{NO}_{3}{ }^{-}\right)$, perchlorate $\left(\mathrm{ClO}_{4}^{-}\right)$, acetate $\left(\mathrm{CH}_{3} \mathrm{COO}^{-}\right)$, sulfate $\left(\mathrm{SO}_{4}{ }^{2-}\right)$
Except $\mathrm{Ba}^{2+}, \mathrm{Hg}^{2+}, \mathrm{Pb}^{2+}$ sulfates
3. Most compounds that contain the following anions are insoluble unless they contain a Group 1A cation, ammonium ion:
Hydroxide $\left(\mathrm{OH}^{-}\right)$, oxide $\left(\mathrm{O}^{2-}\right)$, carbonate $\left(\mathrm{CO}_{3}{ }^{2-}\right)$, phosphate $\left(\mathrm{PO}_{4}{ }^{3-}\right)$, chromate $\left(\mathrm{CrO}_{4}{ }^{2-}\right)$, sulfide $\left(\mathrm{S}^{2-}\right)$
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 solublity 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.

## DONE

