

CHM 191 PRATICAL Manual

EXPERIMENT I

DETERMINATION OF THE AMOUNT OF NaOH AND Na₂CO₃ IN A MIXTURE

1.0 Introduction

In the neutralization reaction of an acid and a base, an indicator is necessary for an end point to be established or for the reaction to occur. For a complete neutralization reaction, the products are usually salt and water only. In our previous knowledge of neutralization reactions, we performed experiments where the reactants dictated the choice of indicators. In particular, only one type of indicator was used for each of the cases and only one reactant was being quantitatively analyzed. However, there are cases where more than one indicator is required for analyzing our sample. That is, the neutralization reaction involving mixed reagents and the use of different indicators in the same titration. This technique becomes useful when we have a mixture of substances making up a solution. In this experiment, we shall perform a neutralization reaction involving mixed bases (base/base) and an acid and also learn how to evaluate the concentration of various components of the mixture at the end of the experiment.

2.0 Objectives

After performing this experiment, students should be able to;

- i. Explain mixed titration
- ii. State the differences between a titration involving single reagent and mixed reagents
- iii. Evaluate the concentration of different components in a mixture
- iv. Learn to use two different types of indicator in one titration
- v. Relate the choice of indicator to type of reaction

3.0 Mixed titration

Mixed titration is a titrimetric process whereby two or more acid/acid or base/base mixture is titrated against a single base or an acid. In such titration, it is necessary to understand all the reactions that take place in the process and be able to write balance equations of all of the reactions without which one cannot evaluate the concentration of the unknown in the mixture. In mixed titration, different types of products are form including intermediate products since the reactions usually involve more than one reaction before the final products are obtained. In

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mixed titration, the polarities of the solvents being mixed together are very important. The two reagents must have affinity for each other. It is always appropriate to use two different indicators in order to be able to detect the end point and establish the transformations that take place from one stage to another with the help of the change in colour of the different indicators. For example, if we have a solution containing a mixture of NaOH and Na₂CO₃, that we wish to analyse, the use of one indicator to signify the end of the reaction would be misleading although an end point was indicated; the two substances could not have each reacted with the acid completely.

Equation for the Reactions



Assuming phenolphthalein is used as indicator, and then the pink colour of the indicator is discharged when reactions 1 and 2 are complete. This is actually the first step of the titration. At this point, the NaOH is neutralized together with half of the Na₂CO₃. The other half of Na₂CO₃ will change to NaHCO₃.

If methyl orange is added and a further quantity of acid is added, the amount of acid required will be that necessary to complete reaction 3. At this stage, the other half of the carbonate is neutralized because one mole of NaHCO₃ is formed from one mole of Na₂CO₃ and hence the quantity of HCl required for reaction 2 and 3 will be the same.

Suppose the volume of the acid needed to reach the end point as indicated by the phenolphthalein is 'x' cm³, and the volume of acid reacting with Na₂CO₃ is '2y' cm³ then the volume of acid reacting with NaOH is $(x + y) - 2y \text{ cm}^3 = (x - y) \text{ cm}^3$

Where:

$y \text{ cm}^3$ = burette reading at methyl orange end point minus burette reading at phenolphthalein end point

$x \text{ cm}^3$ = burette reading at phenolphthalein end point.

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4.0 Materials/Reagents

- i. Burette, 50 cm³
- ii. Pipette, 25 cm³
- iii. Conical flask, 250 cm³
- iv. Volumetric flask/measuring cylinder, 100 cm³
- v. Retort stand with clamp
- vi. Wash bottle
- vii. White paper
- viii. Beaker, 250 cm³
- ix. 'A' is a standard solution of HCl of known molarity. (**Ensure you ask the lab technologist for the molarity**)
- x. 'B' is a solution of two bases containing unknown concentration of NaOH and Na₂CO₃ in the mixture.
- xi. Phenolphthalein indicator
- xii. Methyl orange indicator

5.0 Procedure

First step: Titration of mixture of bases against acid using Phenolphthalein (POP).

- i. Rinse the burette twice with solution 'A' containing HCl and fill it with the same acid. Note the initial burette reading.
- ii. Rinse the pipette with solution 'B' containing a mixture of NaOH and Na₂CO₃ and take 25 cm³ portion of this solution into a 250 cm³ conical flask, add 2-3 drops of Phenolphthalein.
- iii. Add acid from the burette until pink colour disappears. Note the burette reading of the Phenolphthalein.

Second Step: Continuation of titration without adding acid into the burette or base into the conical flask, using methyl orange (MO) as indicator

- iv. Add one drop of methyl orange indicator to the colourless solution obtained at the phenolphthalein end point and continue adding HCl from the burette until one

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drop of acid gives colour change. At this point the remaining NaHCO_3 is neutralized.

- v. Note the burette reading again. This is the methyl orange end point.
- vi. Repeat the titration until three concordant results are obtained.

5.0. Results

Table of Values

Volume of base used = ?

Molar concentration of the acid used = ?

Indicator(s) used = ?

Titration (cm^3)	Rough	1 st Titre	2 nd Titre	3 rd Titre
Second end point (V_2)				
First end point (V_1)				
Initial volume (V_0)				
Volume used ($V_1 - V_0$) POP				
Volume used ($V_2 - V_1$) MO				

Average vol. of acid used for POP = ?

Average vol. of acid used for MO = ?

6.0 Problems

Calculate the following:

- i. The volume of HCl required for complete neutralization of Na_2CO_3
- ii. The volume of HCl required for complete neutralization of NaOH
- iii. The concentration in moles per dm^3 of NaOH in the mixture that has reacted
- iv. The concentration in moles per dm^3 of Na_2CO_3 in the mixture that has reacted.
- v. The amount in grams per dm^3 of the NaOH in the mixture
- vi. The amount in grams per dm^3 of the Na_2CO_3 in the mixture
- vii. The percentage composition of NaOH in the mixture
- viii. The percentage composition of Na_2CO_3 in the mixture

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QUESTIONS

1. What is a mixed titration?
2. Why is it not recommended to use only one type of indicator in neutralization titrations that involve the use of mixed reagents
3. State the differences between a titration involving single reagent and mixed reagents

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EXPERIMENT II

DETERMINATION OF THE CONCENTRATION OF A WEAK ACID IN THE PRESENCE OF A STRONG ACID IN A MIXTURE

1.0 Introduction

In this experiment we will be learning about the titration/neutralization reaction involving a strong acid and a weak acid in mixture being neutralized with a strong base and also learn how to evaluate the concentration of various components of the mixture at the end of the experiment. As we learnt in experiment one (1), two different types of indicators shall be used for an accurate end point or for the complete neutralization of the reaction to form salt and water only.

2.0 Objectives

After performing this experiment, students should be able to:

- i. Explain mixed titration
- ii. State the difference between a titration involving single reagent and mixed reagents
- iii. Evaluate the concentration of different components in a mixture
- iv. Learn to use two different types of indicators in one titration
- v. Relate the choice of indicator to type of reaction

3.0 Mixed Titration

Mixed titration is a titrimetric process whereby two or more acids (acid/acid) or bases (base/base) mixture is titrated against a single base or an acid. In such titration, it is necessary to understand all the reactions that take place in the process and be able to write balance equations of all the reactions without which one cannot evaluate the concentration of the unknown in the mixture. In mixed titration, different types of products are formed including intermediate products since the reactions usually involve more than one reaction before the final products are obtained. In mixed titration, the polarities of the solvents being mixed together are very important. There must be affinity amongst the two reagents. It is always appropriate to use two different indicators in order to be able to detect the end point and

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establish the transformations that take place from one stage to another with the help of the change in colour of the different indicators.

For example, if we have a solution containing a mixture of ethanoic acid (CH_3COOH) and hydrochloric acid (HCl), that we wish to analyze, the use of one indicator to signify the end of the reaction would be misleading although an end point would be indicated, the two substances could not have each reacted with the base completely.

Equations for the Reactions

The equations for the reactions taking place during the reaction of CH_3COOH (weak organic acid) and HCl (strong acid) in a mixture with NaOH (strong base) are as follow:-



Ethanoic acid (CH_3COOH) is a weak organic acid ($K_a = 1.8 \times 10^{-5}$) and is thus not strongly dissociated in water. The reaction that takes place in step I can be monitored using methyl orange as indicator, (methyl orange is a good indicator for strong acid versus strong base titration). The reaction that takes place in step II cannot be monitored with methyl orange, but is detectable with phenolphthalein as indicator. (Phenolphthalein is a good indicator for weak acid versus strong base titration). Thus, if, methyl orange is use as indicator, one can determine the amount of HCl in the mixture. Continuous titration with phenolphthalein indicator is then perform to determine the amount of ethanoic acid in the mixture. The total volume of the base used can also be used to determine the total amount of acids (HCl and CH_3COOH) in the mixture.

The volume of the base (NaOH) consumed to reach end point as indicated by the methyl orange indicator in step I = $x \text{ cm}^3$ and that consumed in step II as indicated by the phenolphthalein indicator is $y \text{ cm}^3$, therefore, the amount of NaOH required to consume the HCl in the mixture is $x \text{ cm}^3$, the amount of NaOH required to consume the total acid in the mixture is y and the amount of NaOH required to consume the $\text{CH}_3\text{COOH} = y-x$.

Where:

$x \text{ cm}^3$ = burette reading at methyl orange end point

$y \text{ cm}^3$ = burette reading at phenolphthalein end point.

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4.0 Procedure

First Step: Titration of mixture of acids against base using methyl orange

- i. Pipette 25 cm³ of the acid mixture into a clean conical flask. Rinse a clean burette with small amount of standard NaOH and fill it with the NaOH above the zero mark, with the aid of a filter funnel. Remove the filter funnel and drain to the zero mark making sure that the burette tip is fill.
- ii. Add 2 or 3 drops of methyl orange indicator to the acid mixture and titrate until the first permanent colour change (orange to yellow).
- iii. Take the reading of the burette and record the volume of base used as x cm³.

Second Step: Continuation of titration without adding base into the burette or acid into the conical flask, using phenolphthalein (POP) as indicator.

- iv. Add 2 or 3 drops of phenolphthalein and continue to titrate until a second colour change is observe.
- v. Take the reading of the burette and record the amount of base used as y cm³
- vi. Repeat your titrations steps (1) – (V) two or more times until concordant results are obtained, record the results as shown below.

5.0 Results

Table of Values

Volume of acid used = ?

Molar concentration of the base used = ?

Indicator (s) used = ?

Burette reading (cm ³)	Rough	1 st titre	2 nd titre	3 rd titre
Second end point (V ₂)				
first end point (V ₁)				
Initial volume (V ₀)				
Volume of base used (V ₁ -V ₀) MO				
Volume of based used (V ₂ -V ₁)POP				
Volume of based used (V ₂ -V ₀) MO & POP				

Average volume of base used for MO = ?

Average volume of base used for POP = ?

Average volume of base used for total acid in the mixture =?

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6.0 Problems

From the average titre values, calculate:

- i. The volume of NaOH required for complete neutralization of HCl in the mixture
- ii. The volume of NaOH required for complete neutralization of the total acid in the mixture
- iii. The volume of NaOH required for complete neutralization of CH₃COOH in the mixture
- iv. The concentration in moles per dm³ of HCl in the mixture
- v. The concentration in moles per dm³ of CH₃COOH in the mixture
- vi. The concentration in grams per dm³ of HCl in the mixture
- vii. The concentration in grams per dm³ of CH₃COOH in the mixture
- viii. The percentage composition of HCl in the mixture
- ix. The percentage composition of CH₃COOH in the mixture
- x. Calculate the pH of the acid mixture.

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EXPERIMENT III

DETERMINATION OF THE PARTITION/DISTRIBUTION COEFFICIENT OF A SOLUTE (IODINE) BETWEEN TWO IMMISCIBLE LIQUIDS/SOLVENTS (CHLOROFORM AND WATER)

1.0 Introduction

In this experiment, the principle of the distribution/partition of a solute (iodine) between two immiscible liquids, otherwise known as partition or distribution coefficient will be discussed and demonstrated. Most commonly, one of the solvents is water (H_2O) while the other is hydrophobic such as chloroform (CHCl_3). Hence, the partition/distribution coefficient measures how hydrophilic ("water-loving") or hydrophobic ("water-fearing") a chemical substance/solute is or the difference in solubility of the solute in the two solvents. Water is known to be a polar molecule/solvent while chloroform is a non-polar solvent.

2.0 Objectives

After studying this guide and having performed the experiment, students should be able to:-

1. Define partition/distribution coefficient and state the mathematical relationship of the distribution/partition of a solute between two immiscible liquids.
2. Demonstrate the distributions/partitions of a solute between two immiscible solvents.
3. Determine the difference in solubility of the solute in the two immiscible solvents.
4. Evaluate the distribution/partition coefficient of a solute between two immiscible solvents, and from a given data.

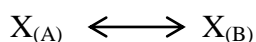
3.0 Theory

If two immiscible solvents A and B are taken in a beaker or a separatory funnel, they form separate layers. When a solute X which is soluble in both solvents is added and shaken, it gets distributed or partitioned between them. Molecules of X pass from solvent A to B and from solvent B to A. Finally, a dynamic equilibrium is set up when the chemical potential (free energies) of the solute in the two solvents are equal provided the molecular state of the solute is the same in each case, that is, there is no association or dissociation. At equilibrium, the rate, at which molecules of X pass from one solvent to the other is balanced. The

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molecular state of iodine in both solvents (CHCl_3 and H_2O) is the same as I_2 . Iodine is soluble both in chloroform and water. In chloroform, it forms a purple coloured solution and in water it is less soluble and forms a brown solution. Hence, the partition coefficient is practically independent of concentration in dilute solutions.

The equilibrium may be expressed as



Where X is the solute partitioned/ distributed between solvents A and B.

At constant temperature and pressure the distribution of the solute between the two solvents may be defined as:-

$$K = \frac{[X]_A}{[X]_B}$$

Where $[X]_A$ and $[X]_B$ are the concentrations of X in A and B respectively. K_D is known as the distribution or partition coefficient. The value of K_D is an indication of the relative solubility of the solute in the two solvents.

4.0 Partition/distribution coefficient

This is the ratio of the concentration of the solute in the two phases of a mixture of two immiscible solvents at a given or constant temperature in a dynamic equilibrium.

In this experiment, the solute is iodine and the solvents are water and chloroform. The molecular state of iodine (I_2) is the same in each solvent and the distribution coefficient is given by;

$$K = \frac{[\text{I}_2]_{\text{CHCl}_3}}{[\text{I}_2]_{\text{H}_2\text{O}}}$$

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5.0 Materials/Reagents

Solid iodine, separatory funnel, conical flasks (250 cm³), distilled water, starch indicator, %10 potassium iodide (KI) solution, sodium thiosulphate solution, pipette and burette.

6.0 Procedure

- i. Weigh 0.7g of solid iodine using a weighing balance into a beaker.
- ii. In a dry stoppered separatory funnel, put 12 cm³ of chloroform, add the accurately weighed 0.7g of solid iodine and add 100 cm³ of distilled water to the separatory funnel. Cover and shake vigorously for 15 –30 minutes until a dynamic equilibrium is established, and allow to stand for 5 – 10 minutes until the phase is completely separated.
- iii. Separate the organic layer from the aqueous layer.
- iv. Pipette 25 cm³ of the aqueous layer into a conical flask, add two drops of starch indicator and titrate with the standard sodium thiosulphate solution until the blue colour disappears.
- v. Transfer 2.5 cm³ of the organic layer into a conical flask, add 10 cm³ of 10 % KI, shake well and titrate with the standard sodium thiosulphate solution until it is pale yellow, then add two drops of starch indicator and continue the titration carefully until the blue colour disappears.

7.0 Calculations

- i. Calculate the concentration of iodine in the aqueous layer
- ii. Calculate the concentration of iodine in the organic layer
- iii. Calculate the partition coefficient

8.0 Conclusion

It is evident that a solute can be soluble in more than one solvent and that the distribution coefficient of a solute between two immiscible liquids is a function of its affinity for the solvents.

9.0 Question

1. Define partition/ distribution coefficient.
2. State the mathematical relationship between the distributions of a solute in the two immiscible liquids.

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3. A solid X is added to a mixture of benzene and water. After shaking well and allowed to stand, 10 cm^3 of the benzene layer was found to contain 0.13 g of X and 100 cm^3 of water layer contained 0.22 g of X. Calculate the value of the distribution coefficient.
4. An aqueous solution of succinic acid at 15°C , containing 0.07 g in 10 cm^3 is in equilibrium with an ethereal solution which has 0.013 g in 10 cm^3 . The acid has its normal molecular weight in both solvents. What is the concentration of the ethereal solution which is in equilibrium with an aqueous solution containing 0.024 g in 10 cm^3 ?
5. At 25°C , an aqueous solution of iodine containing 0.0516 gdm^{-3} is in equilibrium with a carbon tetrachloride (CCl_4) solution containing 4.412 gdm^{-3} . The solubility of iodine in water at 25°C is 0.34 gdm^{-3} . Find the solubility of iodine in carbon tetrachloride.
6. At 25°C the iodine solution in water which contains 0.0516 g lit^{-1} is in equilibrium with CCl_4 solution containing 4.1280 g lit^{-1} of iodine. If at 25°C iodine solution contains 0.410 g lit^{-1} of iodine, find out the solubility of iodine in CCl_4 solution.

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EXPERIMENT IV

SOLUBILITY PROPERTIES OF COMPOUNDS

5.0 Introduction

In this experiment, we shall be looking at one of the basic parameters that require to be considered in every practical analysis, which is solubility of inorganic compounds. Inorganic compounds could be soluble, sparingly soluble and insoluble. The ability of these inorganic compounds to dissolve in water or any given solvents depends on their solubility strength. Some compounds are soluble in particular solvents while others are not. Compound that is soluble in a particular solvent is said to have affinity for such solvent while those that are not soluble in a particular solvent do not have affinity for such solvent. Two or more compounds could be soluble in a particular solvent. Solubility of substances in solvents leads to the formation of solutions. Therefore, solute + solvent = solution, where the substance that dissolves represent the solute and the liquid in which it dissolves represent the solvent.

5.1 Objectives

After studying and having the experiment performed, students should be able to:

- i. Define solubility
- ii. State the solubility of some salts in a given solvents
- iii. List at least five salts that are soluble, sparingly soluble and insoluble in water
- iv. Group salts base on their solubility properties
- v. Differentiate between saturated, unsaturated and supersaturated solution

5.2 Solubility

The **solubility** of a substance is defined as the mass (g) of that substance which will dissolve in a fixed amount of solvent (usually 100 g of liquid) at a given temperature. Depending on its molecular structure, a solute will have different solubility in different solvents.

For Example:

Sodium chloride (table salt) is an ionic compound. It is soluble in polar solvent such as water but insoluble in nonpolar solvents such as cyclohexane and toluene. Sodium chloride may

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also be slightly soluble in a solvent that has polar and nonpolar properties, and such a solvent is a weak polar solvent.

The solubility of a substance in a liquid is temperature dependent. It helps to predict whether a precipitate will form when a compound is added to a solvent or when two solutions are mixed. Solubility is brought about by the introduction of a solute into a solvent to form a partial or clear solution. The substance that is introduced into the solvent is called the **solute** and it is said to be soluble in the **solvent** if it dissolves completely to form a clear solution. The Solution form could be saturated, unsaturated and supersaturated.

A **saturated solution** is that solution which contains the appropriate amount of solute in a given amount of solvent. An **unsaturated solution** is a solution that contains less amount of the solute in a given amount of solvent. It has the capacity to dissolve more solute leaving no undissolved material. On the other hand, a **supersaturated solution** is a solution which contains more than the required amount of solute in a given volume of solvent. The concentration of a solution can be derived by expressing solubility in qualitative terms. For example, the solubility of table salt in water, which is 35.9/100 cm³ of water.

5.3 Solubility of Some Inorganic Salts in Water

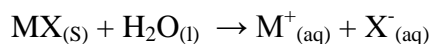
It is observed that some of the salts of a particular metal element are soluble in water while others are sparingly soluble or insoluble in water. The solubility characteristic of some inorganic salts in water is as enumerated below:

1. All common chlorides salts are soluble in water except Lead, Silver and Mercury chlorides
2. All trioxonitrate (V) salts are soluble in water
3. All trioxocarbonate (IV) salts are insoluble except Na, K and NH₄ salts
4. All common tetraoxosulphates (VI) are soluble in water except those of barium, lead and calcium which are sparingly soluble
5. Trioxosulphates (IV) of NH₄, K, Na and Ca are soluble while others are insoluble

All ionic compounds are strong electrolytes, but they are not all equally soluble. A weak electrolyte is a poor conductor of electricity while a strong electrolyte is a good conductor of electricity.

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For ionic compounds, an equation which reflects the solubility behaviour of a solution can be written. If the compound is soluble, the equation is written as:



When the solid ionic compound MX is added to water, it dissolves forming the aqueous ions M^+ and X^- . The presence or absence of ions can be determined by conductivity. If the compound is insoluble in water the equation can be written as:



5.4 Materials/Reagents

- i. Conical flask, 250 cm³
- ii. Volumetric flask/measuring cylinder, 100 cm³
- iii. Wash bottle
- iv. Beaker, 250 cm³
- v. Distilled water
- vi. Test tube
- vii. Different inorganic salts (A, B, C, D, E)

5.5 Procedure

- i. Rinse the test tube, measuring cylinder and conical flask with distilled water.
- ii. Put the salts in separate test tubes and label as A, B, C, D and E
- iii. To A and B add 3 cm³, C and D add 2 cm³ and E add 10 cm³ of water respectively.
- iv. Observe the solubility and colour of the solution.
- v. Record the observation in the table below

Test	Colour of Solid	Solubility in Water	Colour of Solution
A			
B			
C			
D			
E			

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Questions

1. Using water as solvent, classify the following compounds as soluble, sparingly soluble and insoluble

Salt	Soluble	Insoluble	Sparingly Soluble
Ag_2SO_4			
CaCO_3			
Na_3PO_4			
$\text{Zn}(\text{NO}_3)_2$			

2. Define solubility
3. Distinguish between the terms slightly soluble and weak electrolyte.
4. When is a solution said to be saturated, unsaturated and supersaturated?
5. List ten salts each that are soluble, sparingly soluble and insoluble in water