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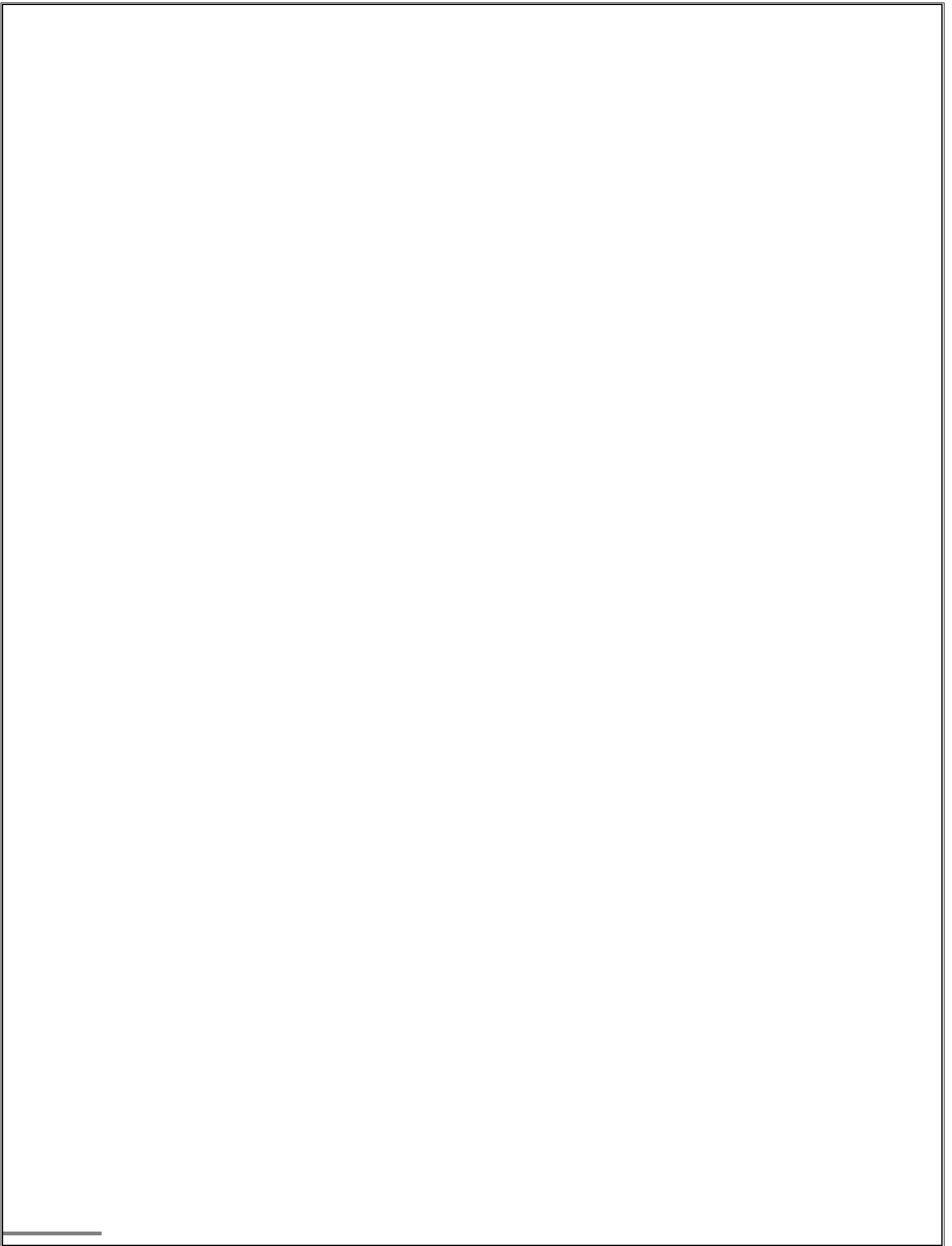

REGULATIONS-2021

BS3171 –CHEMISTRY LABORATORY

LABORATORY MANUAL

INDEX

DETERMINATION OF TOTAL, TEMPORARY AND PERMANENT HARDNESS OF WATER BY EDTA METHOD

Short Procedure:

Titration I: **Standardization of EDTA**

Burette Solution : **EDTA Solution**

Pipette Solution : **Standard Hard Water**

Reagents added : **5 ml of ammonia buffer**

Indicator : **Eriochrome black – T**

End Point : **Colour change from wine red to clear blue**

Standard Hard Water Vs EDTA

| S.No | Volume of Standard Hard Water (ml) | Burette Reading (ml) | | Volume of EDTA Solution (ml) |
		Initial	Final	
1.	20			
2.	20			

Concordant Value: ml

Calculation:

Volume of standard hard water $V_1 = 20$ ml

Strength of standard hard water $N_1 = $ --------- N

Volume of EDTA $V_2 = $ -------- ml

Strength of EDTA $N_2 = $

According to the law of volumetric analysis $V_1 N_1 = V_2 N1$

$$N_2 = \frac{V_1 N_1}{V_2}$$

Strength of EDTA, $N_2 = $ ----- N

Expt.No: 01	DETERMINATION OF TOTAL, TEMPORARY AND PERMANENT
Date:	HARDNESS OF WATER BY EDTA METHOD

AIM

  To determine the total, temporary and permanent hardness in the given sample of hard water by EDTA method. Standard hard water (0.015 N) and EDTA solution are provided.

PRINCIPLE

  Hard water does not given lather freely with soap. Such water form precipitate with soap. Water is said to be hard when it contains Ca^{2+}, Mg^{2+} or any other heavy metal ion (other than alkali metal ion), Disodium salt of Ethylene Diamine Tetra Acetic acid (EDTA) is a well know complexing agent. Its structure is shown below

$$NaOOCCH_2 \qquad CH_2COOH$$
$$\backslash \qquad /$$
$$NCH_2CH_2N$$
$$/ \qquad \backslash$$
$$HOOOCCH_2 \qquad CH_2COONa$$
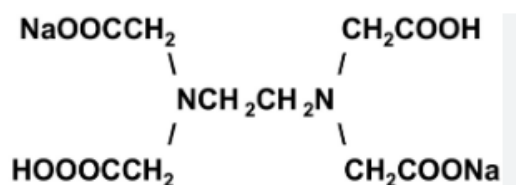
Fig: Disodium salt of Ethylene Diamine Tetra Acetic acid (EDTA)

  Disodium salt of EDTA is used to estimate the various hardness of the given hard water containing Ca^{2+} and Mg^{2+} ions. When EDTA is added to hard water, it reacts with calcium and magnesium ions present in hard water to form stable EDTA metal complexes. From the volume of EDTA consumed the hardness can be calculated. Eriochrome Black-T is used as an indicator. The indicator forms a weak complex with the metal ions present in the hard water and gives wine red colour.

$$\begin{pmatrix} Ca^{2+} \\ Mg^{2+} \end{pmatrix} + EBT \xrightarrow{pH = 9\text{-}10} \begin{pmatrix} Ca^{2+} \\ Mg^{2+} \end{pmatrix} \text{EBT complex}$$

Hard water   Indicator   (Weak) (Wine red colour)
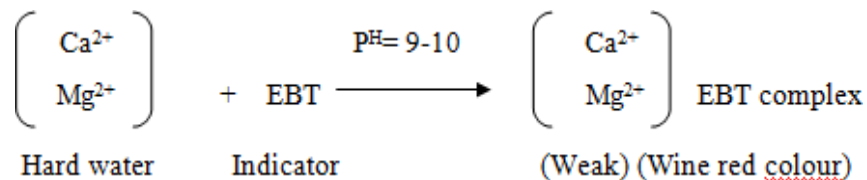
Materials Required

  EDTA, Ammonium chloride, Ammonium hydroxide, Eriochrome Black-T, Calcium carbonate, Standard hard water, Sample hard water, Burette, Pipette, Conical flask.

Short Procedure:

Titration II: Determination of Total Hardness of Hard Water Sample

Burette Solution	**: EDTA Solution**
Pipette Solution	**: Hard water sample**
Reagents added	**: 5 ml of ammonia buffer**
Indicator	**: Eriochrome black – T**
End Point	**: Colour change from wine red to clear blue**

HARD WATER SAMPLE Vs EDTA

S.No	Volume of hard water sample (ml)	Burette Reading (ml)		Volume of EDTA Solution (ml)
		Initial	**Final**	
1.	20			
2.	20			

Concordant Value: ml

Calculation:

Volume of EDTA $V_1 = \text{-------} ml$

Strength of EDTA $N_1 = \text{------} N$

Volume of hard water sample $V_2 = 20\ ml$

Strength of hard water sample $N_2 = \text{---------}\ N$

According to the law of volumetric analysis $V_1 N_1 = V_2 N_2$
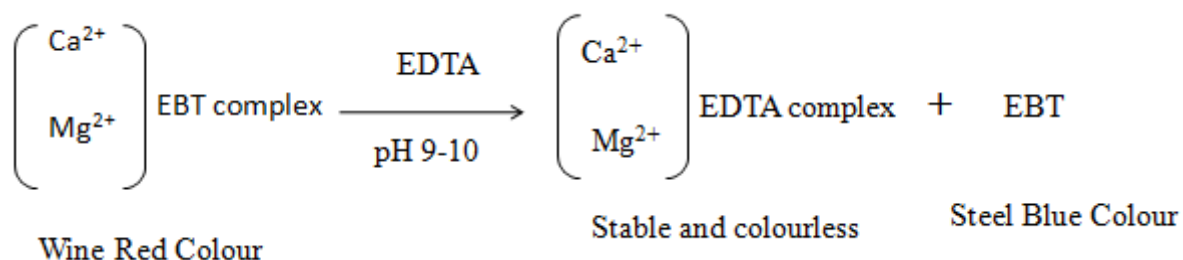
$$N_2 = \frac{V_1 N_1}{V_2}$$

$$N_2 = \underline{\qquad} N$$

Total hardness of the given sample of hard water   = --------- 50 x1000

                                                   = ...ppm.

When EDTA is added into the hard water, the metal ions form a stable metal complex with EDTA by leaving the indicator. When all the metal ions are taken by EDTA from the indicator metal ion complex, the wine red colour changes into steel blue, which denotes the end point. The metal EDTA complex is stable at p^H 8-10. This p^H range can be maintained by adding ammoniacal buffer (NH_4Cl + NH_4OH).

$$\begin{bmatrix} Ca^{2+} \\ \\ Mg^{2+} \end{bmatrix} \text{EBT complex} \xrightarrow[\text{pH 9-10}]{\text{EDTA}} \begin{bmatrix} Ca^{2+} \\ \\ Mg^{2+} \end{bmatrix} \text{EDTA complex} + \text{EBT}$$

Wine Red Colour   Stable and colourless   Steel Blue Colour

PROCEDURE:

Titration I: Standardization of EDTA

The burette is washed with distilled water and rinsed with a little amount of given EDTA solution and filled with the same up to zero mark. 20ml of standard hard water solution is pipetted out into a clean conical flask. 5 ml of ammonia buffer solution and a pinch of Eriochrome Black –T indicator are added. The solution turns wine red in colour. It is then titrated against EDTA taken in the burette. The end point is change in colour from wine red to steel blue. The reading is noted. The titration is repeated to get concordant values.

Titration II: Determination of Total hardness of hard water sample.

20 ml of the given hard water sample is pipetted out into a clean conical flask.5 ml of ammonia buffer solution and a pinch of Eriochrome Black- T indicator is added. The solution turns wine red in colour. This solution is titrated against EDTA solution taken in the burette. The end point is change in colour from wine red to steel blue. The titration is repeated to get concordant values.

Short Procedure:

Titration III: Determination of Permanent Hardness

Burette Solution : **EDTA Solution**

Pipette Solution : **Boiled hard water sample**

Reagents added : **5 ml of ammonia buffer**

Indicator : **Eriochrome black – T**

End Point : **Colour change from wine red to clear blue**

Equivalent weight of $CaCO_3$ = 50

Boiled hard water sample Vs EDTA

S.No	Volume of boiled hard water sample (ml)	Burette Reading (ml)		Volume of EDTA Solution (ml)
		Initial	Final	
1	20			
2	20			

Concordant Value:　　ml

Calculation:

Volume of EDTA $\qquad$ V_1 = ------ ml

Strength of EDTA $\qquad$ N_1 =--------- N

Volume of boiled hard water sample $\qquad$ V_2 = 20 ml

Strength of boiled hard water sample $\qquad$ N_2 =-----------N

According to the law of volumetric analysis $\qquad$ $V_1 N_1 = V_2 N_2$

$$N_2 = \frac{V_1 N_1}{V_2}$$

=

=-----------60x 1000

Permanent hardness of the given sample of hard water $\quad$ =-------ppm.

Determination of Temporary Hardness:
Temporary hardness of the given sample of water $\quad$ = Total hardness – Permanent hardness

= ---------- ....ppm. =...........ppm.

Titration –III: Determination of Permanent Hardness

      20 ml of the given boiled hard water sample is pipetted out into a clean conical flask.5 ml of ammonia buffer solution and a pinch of Eriochrome Black- T indicator are added. The solution turns wine red in colour. This solution is titrated against the EDTA taken in the burette. The end point is change in colour from wine red to steel blue. The titration is repeated to get concordant values.

Determination of Temporary Hardness

      Temporary hardness of the water sample is calculated by subtracting permanent hardness from total hardness.

RESULT:

    1. Total hardness of the given sample of water   =_____ppm.

    2. Permanent hardness of the given sample of water   =_____ppm.

    3. Temporary hardness of the given sample of water   =_____ppm.

ESTIMATION OF COPPER CONTENT OF THE GIVEN SOLUTION BY IODOMETRY

Short Procedure:

Titration I: Standardization of Sodium thiosulphate

Burette solution   :   Sodium thiosulphate

Pipette solution   :   Potassium dichromate

Reagent added   : 10 ml of dil.H_2SO_4+10 ml of 10% KI

Indicator   :   1 ml Starch

End point   : Disappearance of blue colour

Standard Potassium dichromate V$_s$ Sodium thiosulphate

S.No	Volume of potassium dichromate (ml)	Burette Reading (ml)		Volume of sodium thiosulphate (ml)
		Initial	**Final**	
1.	20			
2.	20			

Concordant Value =   ml

Calculation

Volume of potassium dichromate $V_1 =$ 20 ml

Strength of potassium dichromate N_1 = --------- N

Volume of sodium thiosulphate V_2 =--------- ml

Strength of sodium thiosulphate N_2 =

According to the law of volumetric analysis $V_1 N_1 = V_2 N_2$

$$N_2 = \frac{V_1 N_1}{V_2}$$

 Strength of sodium thiosulphate   = ---------N

Expt.No:02	ESTIMATION OF COPPER CONTENT OF THE GIVEN SOLUTION
Date:	BY IODOMETRY

AIM

To estimate the amount of copper present in the given solution by iodometric titration. A standard solution of   N $K_2Cr_2O_7$ solution is provided.

PRINCIPLE

Copper ion occurs naturally in drinking water and is a micronutrient required for the metabolism of living beings. But the presence of copper in water, in quantities more than1.3mg/lit, will cause stomach ache, intestinal distress and digestive problems. High concentration of copper will also impart a metallic bitter taste to water. Occurrence of copper ions in drinking water may be due to corrosion in plumbing materials and faulty water treatment processes. Copper is also known to cause toxicity to aquatic organism. Titrimetric estimation of copper is done through a redox reaction in which stoichiometric quantity of iodine is liberated on reaction with potassium iodide. The liberated iodine can be titrated against standardized sodium thiosulphate solution.

When KI is added to the copper ion solution, copper ions react with KI liberates I_2.

$$2 CuSO_4 + 4 KI \longrightarrow 2 K_2SO_4 + 2 CuI + I_2$$

The liberated I_2 is titrated against sodium thiosulphate, using starch indicator.

$$2Na_2S_2O_3 + I_2 \longrightarrow 2NaI + Na_2S_4O_6$$
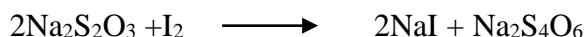
                                        Sodium tetrathionate

$$2Cu^{2+} \equiv I_2 \equiv 2Na_2S_2O_3$$
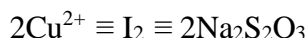
Materials Required

Potassium dichromate, Sodium thiosulphate, Potassium iodide, Starch, Sulphuric acid, Ammonia, Acetic acid, Ammonium hydroxide

Short Procedure:

<u>Titration II:</u> Estimation of copper content of the given solution

Burette solution   :   Sodium thiosulphate

Pipette solution   : Given copper ion

Reagent added   :   10 ml of dil.H_2SO_4+10 ml of 10% KI

Indicator   :   1 ml Starch

End point   :   Disappearance of blue colour

Equivalent weight of copper : 63.5

<div align="center">

Given copper ion V_s Sodium thiosulphate

</div>

S.No	Volume of given copper ion (ml)	Burette Reading (ml)		Volume of sodium thiosulphate (ml)
		Initial	**Final**	
1.	20			
2.	20			

Concordant Value =   ml

Calculation

Volume of sodium thiosulphate $V_1 =$ …… ml

Strength of sodium thiosulphate $N_1 =$----------------N

Volume of the given copper ion $V_2 =$ 20 ml

Strength of the given copper ion $N_2 =$

}According to the law of volumetric analysis $V_1 N_1 = V_2 N_2$

$$N_2 = \frac{V_1 N_1}{V_2}$$

Strength of the given copper ion   = ----------- N

Amount of copper ion present   =   Equivalent weight of copper x

 in the given solution   Strength of given copper ion

                                              =   63.5 x -----------

                                              = --------------------gms / lit

Titration I

Standardization of sodium thiosulphate

20 ml of standard potassium dichromate solution is pipetted out into a clean conical flask. About 10 ml of dilute H_2SO_4 and 10 ml of 10% KI are added to it. The liberated iodine is immediately titrated against sodium thiosulphate solution taken in the burette. When the solution turns pale yellow, about 1 ml of freshly prepared starch is added and the titration is continued. The end point is the disappearance of blue colour. Titration is repeated for concordant values.

TITRATION II

Estimation of copper ion content of the given solution

20 ml of the given copper ion solution is pipetted out into a clean conical flask. About 10ml of dil. H_2SO_4 and 10ml of 10% KI solutions are added to this solution and the liberated iodine is titrated against standardized sodium thiosulphate taken in the burette. When the solution turns pale yellow, about 1ml of freshly prepared starch indicator is added and the titration is continued. The disappearance of blue colour is the end point. The titration is repeated for concordant values.

RESULT

Strength of the given copper ion solution   =-----------N

The amount of copper ion present in the given solution = ... gms/lit

DETERMINATION OF TYPES AND AMOUNT OF ALKALINITY IN WATER SAMPLE

Short Procedure:

Titration I: **Standardization of HCl**

Burette solution **: HCl**

Pipette solution **: NaOH**

Indicator **: Phenolphthalein**

End point **: Disappearance of pink colour**

NaOH Vs HCl

S.No	Volume of NaOH (ml)	Burette Reading (ml)		Volume of HCl (ml)
		Initial	**Final**	
1.	20			
2.	20			

Concordant Value =   ml

Volume of NaOH	V_1= 20 ml
Strength of NaOH	N_1 =----------N
Volume of HCl	V_2 = --------ml
Strength of HCl	N_2 =
According to volumetric law	$V_1N_1=V_2N_2$

$$N_2 = \frac{V_1N_1}{V_2}$$

Strength of HCl   = ------- N

Expt.No: 03	DETERMINATION OF TYPES AND AMOUNT OF
Date:	ALKALINITY IN WATER SAMPLE

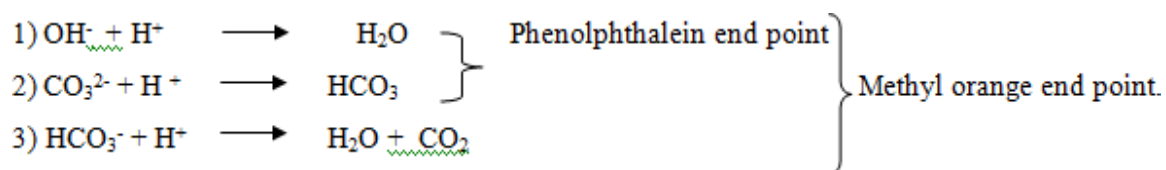**AIM:**

To determine the type and amount of alkalinity present in the given water sample. A standard solution of sodium hydroxide of strength ---------- is given.
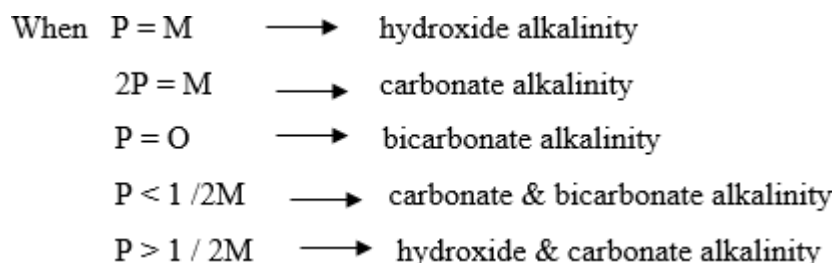
PRINCIPLE:

Alkalinity is caused by the presence of hydroxide, carbonate and bicarbonate. There are five alkalinity conditions possible in a given sample of water, hydroxide only, carbonate only, bicarbonate only, combination of carbonate and hydroxide or carbonate and bicarbonate.

The various alkalinities can be estimated by titrating with a standard acid using phenolphthalein and methyl orange indicators successively.

1) $OH^- + H^+ \longrightarrow H_2O$ Phenolphthalein end point

2) $CO_3^{2-} + H^+ \longrightarrow HCO_3$ Methyl orange end point.

3) $HCO_3^- + H^+ \longrightarrow H_2O + CO_2$

When alkaline water is titrated with acid, phenolphthalein is added. Pink colour disappears when the reactions 1) & 2) are over. Then methyl orange is added and it will change its colour after all three reactions are over. Hence methyl orange end point corresponds to total alkalinity. Let P corresponds to phenolphthalein end point and M corresponds to methyl orange end point.

When P = M $\longrightarrow$ hydroxide alkalinity

       2P = M $\longrightarrow$ carbonate alkalinity

       P = O $\longrightarrow$ bicarbonate alkalinity

       P < 1/2M $\longrightarrow$ carbonate & bicarbonate alkalinity

       P > 1/2M $\longrightarrow$ hydroxide & carbonate alkalinity

Alkalinity values are expressed in terms of milligram per liter as $CaCO_3$.

Materials Required

Hydrochloric acid, Water sample, Sodium hydroxide, Sodium carbonate, Phenolphthalein, Methyl orange, Burette, Pipette, Conical flask

Short Procedure:

<u>Titration II</u> : Determination of alkalinity in water sample

Burette solution   : Hydrochloric acid

Pipette solution   : Water sample

Indicator   : i) Phenolphthalein

   ii) Methyl orange

End point   : i) Disappearance of pink colour

   ii) Colour change from yellow to red orange

Equivalent weight of CaCO$_3$ =50

Water sample Vs standard HCl

S.No	Volume of water sample (ml)	Volume of HCl (ml)	
		Phenolphthalein end point[P]	**Methyl orange end point**[M]
1.	20		
2.	20		

Concordant Value: [P] =   ml   [M]=   ml

Calculation:

   If the data satisfies the condition P >1/2 M, alkalinity is due to hydroxide and carbonate. Bicarbonate is not present.

i) Volume of standard HCl required for [OH$^-$] alkalinity   = 2[P] – [M]

=...............

=............ml

ii) Volume of standard HCl required for [CO$_3^{2-}$] alkalinity   = 2[M] – 2[P]

= ....

=............ml

iii) HCO$_3^-$ alkalinity is not present.

Alkalinity conditions

S.No	Result of titration [P] and [M]	Hardness causing ions		
		OH⁻	CO₃²⁻	HCO₃⁻
1	[P]=0	0	0	[M]
2	[P]=[M]	[P]or[M]	0	0
3	[P] =1/2[M]	0	2[P] or [M]	0
4	[P] > 1/2[M]	2[P] – [M]	2[M] – 2[P]	0
5	[P] < 1/2[M]	0		{[M] – 2[P]}

PROCEDURE:

TITRATION I: Standardization of HCl

The burette is washed with distilled water and rinsed with the given hydrochloric acid. It is filled with the same hydrochloric acid upto zero mark. Pipette out 20 ml of standard NaOH solution in a clean conical flask. Add 2 drops of phenolphthalein indicator into it. The pink coloured solution is titrated against HCl taken in the burette. The end point is the disappearance of pink colour. Titre value is noted and the titrations are repeated for concordant values.

TITRATION II: Determination of alkalinity

The burette solution is the same HCl solution. 20ml of water sample is pipetted out in to a clean conical flask. A drop of phenolphthalein indicator is added. Pink colour is observed. This solution is titrated against the standard acid until pink colour disappeared. The end point is noted. This titre value corresponds to phenolphthalein end point (P).

Few drops of methyl orange indicator is added to the same solution after the phenolphthalein end point. The titration is continued until the solution becomes red orange. The total titre value is noted. This titre value corresponds to methyl orange end point (M). The titration is repeated for concordant values.

From the titre values the amount of each alkalinity present in the given water sample is calculated.

Calculation of OH⁻ alkalinity:

Volume of HCl V_1 = ------- ml

Strength of HCl N_1 = ---------N

Volume of water sample V_2 = 20 ml

Strength of water sample due to OH⁻ alkalinity N_2 =

According to the law of volumetric analysis $V_1 N_1 = V_2 N_2$

$$N_2 = \frac{V_1 N_1}{V_2}$$

= ....N

Amount of OH⁻ content present in 1 litre of water $\left.\right\}$ = Strength of water sample x

sample in terms of $CaCO_3$ equivalent   Equivalent weight of $CaCO_3$

OH⁻ alkalinity in terms of $CaCO_3$ equivalent   = ---------- x50 x 1000

= ....ppm.

Calculation of CO_3^{2-} alkalinity:

Volume of HCl V_1 = ------- ml

Strength of HCl N_1 = ---------N

Volume of water sample V_2 = 20 ml

Strength of water sample due to CO_3^{2-} alkalinity N_2 =

According to the law of volumetric analysis $V_1 N_1 = V_2 N_2$

$$N_2 = \frac{V_1 N_1}{V_2}$$

= ....N

Amount of CO_3^{2-} content present in 1 litre of water $\left.\right\}$ = Strength of water sample x

sample in terms of $CaCO_3$ equivalent   Equivalent weight of $CaCO_3$

= ....x 50

CO_3^{2-} alkalinity in terms of $CaCO_3$ equivalent   = ……... x1000

= ....ppm.

RESULT:

The given water sample contains the following alkalinity

 i) Hydroxide alkalinity   = -------- ppm

ii) Carbonate alkalinity   = ------ ppm

iii) Total alkalinity   = ------- ppm

DETERMINATION OF CHLORIDE CONTENT OF WATER SAMPLE BY ARGENTOMETRIC METHOD

Short Procedure:

Titration I **Standardization of Silver nitrate**

Burette solution :   Silver nitrate

Pipette solution   :   Sodium chloride

Indicator   :   1 ml of 2% Potassium chromate

End point   :   Colour change from yellow to reddish brown

Standard Sodium chloride V_s Silver nitrate

S.No	Volume of Sodium chloride (ml)	Burette Reading (ml)		Volume of Silver nitrate (ml)
		Initial	Final	
1.	20			
2.	20			

Concordant Value =   ml

Calculation

Volume of sodium chloride $V_1 = 20$ ml

Strength of sodium chloride N_1 = --------- N

Volume of silver nitrate V_2 = -------- ml

Strength of silver nitrate $N_2 =$

According to the law of volumetric analysis $V_1N_1 = V_2N_2$

$$N_2 = \frac{V_1N_1}{V_2}$$

$$=$$

 Strength of silver nitrate   = ---------N

| Expt.No:04	**DETERMINATION OF CHLORIDE CONTENT OF WATER**
Date:	**SAMPLE BY ARGENTOMETRIC METHOD**

AIM

To determine the amount of chloride ion present in whole of the given water sample by argentometric method. A standard solution of sodium chloride of strength N and an approximately -------N solution of silver nitrate is provided.

PRINCIPLE

Natural water contains Cl^- ion in the form of NaCl, KCl, $CaCl_2$ and $MgCl_2$.The total Cl^- ion can be estimated by argentometric method. It is known as Mohr's titration. Hence Cl^- ion solution is titrated with $AgNO_3$ using potassium chromate (K_2CrO_4) as the indicator. It is a precipitation titration.

$$AgNO_3 + NaCl \longrightarrow AgCl \downarrow + NaNO_3$$
$$(in\ water) \qquad (white\ precipitate)$$
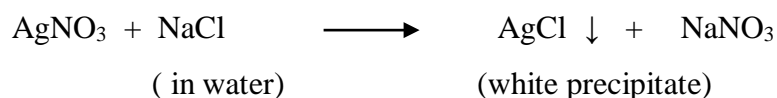
When all the Cl^- ion is removed, $AgNO_3$ added from the burette will react with K_2CrO_4 to give a reddish brown colour due to the formation of Ag_2CrO_4. This is the end point. This reaction takes place only at the end point, since a precipitation reaction takes place in preference.

$$2\ AgNO_3 + K_2CrO_4 \longrightarrow Ag_2CrO_4 + 2KNO_3$$
$$(yellow) \qquad (reddish\ brown)$$
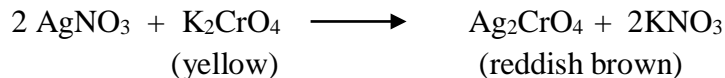
MATERIALS REQUIRED

Sodium chloride, Silver nitrate, Potassium chromate, Water sample, Burette, Pipette, Conical flask

PROCEDURE

TITRATION I: Standardization of Silver nitrate

The burette is washed with distilled water and rinsed with the given silver nitrate solution. It is filled with the same silver nitrate solution upto zero mark. 20ml of the water sample is pipetted out into a clean conical flask.1ml of K_2CrO_4 solution is added. It is then titrated with silver nitrate solution in the burette. The end point is the change of colour from yellow to reddish brown. Titre value is noted and the titrations are repeated for concordant values.

Short Procedure:

<u>TITRATION II</u>: Determination of chloride ion

Burette solution	:	**Silver nitrate**
Pipette solution	:	**Water sample**
Indicator	:	**1 ml of 2% Potassium chromate**
End point	:	**Colour change from yellow to reddish brown**
Equivalent weight of chloride	:	**35.46**

Water sample V_s Silver nitrate

S.No	Volume of water sample (ml)	Burette Reading (ml)		Volume of Silver nitrate (ml)
		Initial	Final	
1.	20			
2.	20			

Concordant Value =   ml

Calculation

Volume of silver nitrate	V_1 = -------- ml
Strength of silver nitrate	N_1 = --------- N
Volume of the water sample	V_2 = 20 ml
Strength of the water sample	N_2 =
According to the law of volumetric analysis	$V_1 N_1 = V_2 N_2$
	$N_2 = \dfrac{V_1 N_1}{V_2}$
Strength of the water sample	= ----------- N

Calculation of amount of the chloride ion

Amount of the chloride ion present in 1litre of the water sample	=	Equivalent weight of chloride ion x Strength of chloride ion
	=	35.46 x---------- 1000 mgs/lit
	=	------------mgs / lit (or) ppm

TITRATION II: Estimation of chloride ion

    20 ml of the given water sample is pipetted out into a clean conical flask and 1 ml of K_2CrO_4 solution is added. It is then titrated against standardized silver nitrate solution taken in the burette. The end point is the change of colour from yellow to reddish brown. Titre value is noted and the titrations are repeated for concordant values.

RESULT

    Amount of chloride ion present in1 litre of the water sample   = ----------mgs / lit (or) ppm.

DETERMINATION OF STRENGTH OF GIVEN HYDROCHLORIC ACID USING pH- METER

Short Procedure

Burette solution   **:**   **Sodium Hydroxide**

Pipette solution   **:**   **20 ml of HCl + 30 ml of distilled water**

Place the combined electrode in the beaker solution and pH is noted

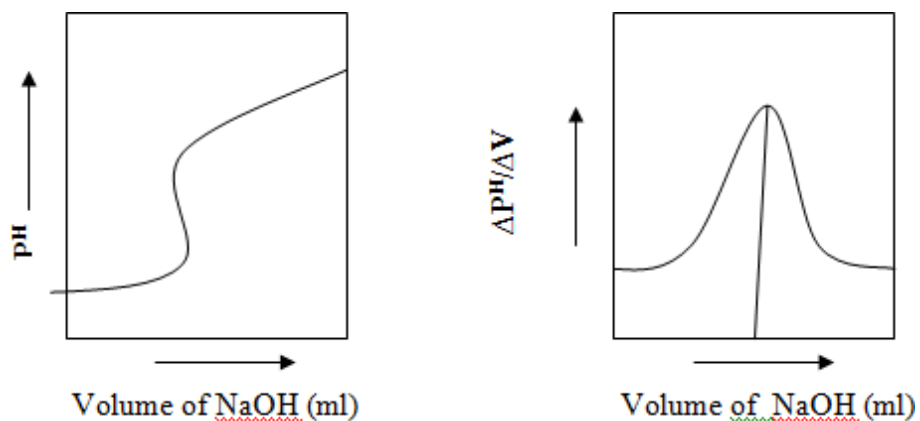A graph is plotted between volume of NaOH in x axis and pH in y axis

To find accurate end point, a graph is plotted between volume of NaOH in x axis and Δ pH /ΔV in y axis

From the graph volume of NaOH is obtained

Equivalent weight of HCl = 36.45

The amount of HCl = Strength of HCl x Equivalent weight of HCl

Model Graph

| Expt.No:05	**DETERMINATION OF STRENGTH OF GIVEN HYDROCHLORIC ACID USING pH – METER**
Date:	

AIM

To determine the strength of given HCl using pH-meter, a standard solution of NaOH of strength -------is provided.

PRINCIPLE

The P^H of the solution is related to $[H^+]$ ion concentration by the formula,

$$pH = - \log [H^+]$$

Measurement of pH of the solution gives the concentration of H^+ ions in the solution. When NaOH is added slowly from the burette to the solution of HCl, the fast moving H^+ ions are progressively replaced by slow moving Na^+ ions. As a result pH of the solution increases.

$$HCl + NaOH \rightarrow NaCl + H_2O$$
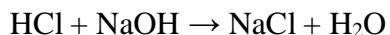
The increase in pH takes place until all the H^+ ions are completely neutralized (up to the end point). After the end point, further addition of a NaOH increases pH sharply as there is an excess of fast moving OH^- ions.

MATERIALS REQUIRED:

pH meter, Glass electrode, Beaker, Standard NaOH, Given HCl solution, Burette, Pipette, etc.

Titration of HCl Vs NaOH

S.No	Volume of NaOH (ml)	pH	Δ pH	ΔV(ml)	$\Delta p^H/\Delta V$

PROCEDURE:

The burette is cleaned with distilled water and filled with NaOH solution upon the mark. 20 ml of the given HCl is pipetted out into a clean 100 ml beaker and 30 ml of distilled water is added. The glass electrode is dipped in it and connected with a pH meter.

The pH of the solution is measured for HCl alone (without the addition of NaOH). Now 1ml of NaOH from the burette is added to the solution taken in the beaker, stirred for some time and the pH is noted. The process is continued by adding 1ml of NaOH each time. At least five readings are taken even after the end point.

A graph is plotted by taking volume of NaOH in x-axis and pH in y-axis. Accurate end point is determined by plotting volume of NaOH in the X-axis and $\Delta pH/\Delta V$ in y-axis. From the end point, strength of the HCl solution and hence the amount can be determined.

Calculation

I) To calculate the strength of HCl

Volume of HCl $\qquad$ $V_1 = 20$ ml

Strength of HCl $\qquad$ $N_1 =$

Volume of NaOH $\qquad$ $V_2 =$ ------ ml

Strength of NaOH $\qquad$ $N_2 =$ ......N

According to the law of volumetric analysis, $V_1N_1 = V_2N_2$

$$N_2 = \frac{V_1N_1}{V_2}$$

Strength of HCl $\qquad$ = ------ N

II) To calculate the amount of HCl

The amount of HCl present in 1000 ml of the given solution $\qquad$ = Strength of HCl x Equivalent mass of HCl

$$= \text{---------} \times 36.45$$

$$= \text{-------} \text{ gms.}$$

RESULT:

i)   Strength of the given HCl solution   =------- N

ii)   Amount of HCl present in 1000 ml of the given solution = --------gms.

CONDUCTOMETRIC TITRATION OF BARIUM CHLORIDE AGAINST SODIUM SULPHATE (PRECIPITATION TITRATION)

Short Procedure:

Burette solution   :   Sodium sulphate

Pipette solution   :   20 ml of Barium chloride + 30 ml distilled water

Place the conductivity cell in the beaker solution and conductance noted.

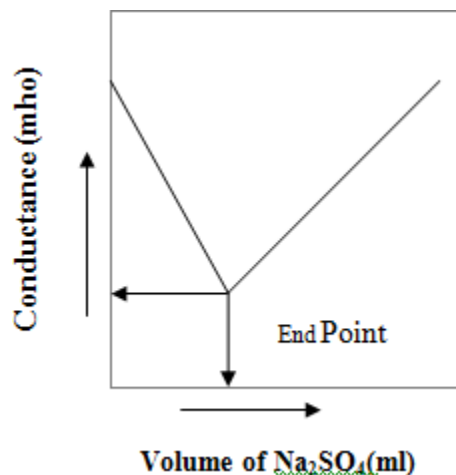A graph is plotted between volume of Na_2SO_4 in x axis and conductance in y axis.

The conductance decreases and then increases

From the graph volume of Na_2SO_4 is obtained

Equivalent weight of $BaCl_2$ =122.14

The amount of $BaCl_2$= Strength of $BaCl_2$ x Equivalent weight of $BaCl_2$

Model Graph

Volume of Na₂SO₄(ml)

| Expt.No:06	**CONDUCTOMETRIC TITRATION OF BARIUM CHLORIDE**
Date:	**AGAINST SODIUM SULPHATE (PRECIPITATION TITRATION)**

AIM:

To determine the amount of $BaCl_2$ present in the given solution by conductometric titration using standard Na_2SO_4 of strength ------- N

PRINCIPLE:

Solutions of electrolytes conduct electricity due to the presence of ions. Since specific conductance of a solution is proportional to the concentration of ions in it, conductance of the solution is measured during titration.

In the precipitation titration, the ions are converted into insoluble precipitate, which will not contribute to the conductance. $BaCl_2$ reacts with Na_2SO_4 to given $BaSO_4$ precipitate.

$BaCl_2 + Na_2SO_4 \rightarrow BaSO_4\downarrow + 2NaCl$
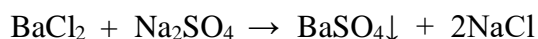
Unionized

The Ba^{2+} ions in the solution are replaced by free Na^+ ions. Since the mobility of Na^+ ions are less than that of Ba^{2+} ions, the conductance of the solution decreases. After the end point when all the Ba^{2+} ions are replaced, further addition of Na_2SO_4 increases the conductance. This is due to the increase of Na^+ and SO_4^{2-} ions in the solution.

MATERIALS REQUIRED:

Conductivity meter, Conductivity cell, Beaker, Standard Na_2SO_4 solution, Given $BaCl_2$ solution, Burette, Pipette.

Titration of BaCl$_2$V$_S$ Na$_2$SO$_4$

S.No	Volume of Na$_2$SO$_4$ added(ml)	Conductance (mho)

PROCEDURE:

The burette is cleaned with distilled water and filled with Na_2SO_4 solution upto the mark. 20ml of the given $BaCl_2$ solution is pipetted out into a clean 100 ml beaker. 30 ml of distilled water is added. The conductivity cell is placed in it such that the electrodes are well immersed in the solution. The cell is connected to the conductivity meter.

The conductance is read from the bridge for the $BaCl_2$ solution alone (without the addition of Na_2SO_4). Now 1 ml of Na_2SO_4 from the burette is added to the solution taken in the beaker, stirred for some time and the conductance is measured. The conductivity goes on decreasing upto the end point. At least five readings are taken after the end point has been reached.

Now a graph is plotted by taking volume of Na_2SO_4 in the X – axis and conductance in the Y-axis. The end point is noted. It is the intersection of the two lines. From the end point the amount of $BaCl_2$ present in the given solution is calculated.

Calculation:

i) Calculation of strength of $BaCl_2$:

Volume of $BaCl_2$	$V_1 = 20$ ml
Strength of $BaCl_2$	$N_1 =$
Volume of Na_2SO_4	$V_2 =$ ------ml
Strength of Na_2SO_4	$N_2 =$ ------ N
According to the law of volumetric analysis,	$V_1N_1 = V_2N_2$

$$N_2 = \frac{V_1N_1}{V_2}$$

Strength of $BaCl_2$ = ------N

ii) Calculation of amount of $BaCl_2$:

The amount of $BaCl_2$ present in 1000 ml   = Strength of $BaCl_2$ x Equivalent mass

of the given solution of $BaCl_2$

                                       = --------- x122.14

                                       = --------gms.

RESULT:

The strength of $BaCl_2$ solution =------- N.

The amount of $BaCl_2$ present in the given solution = -------gms.

DETERMINATION OF STRENGTH OF ACIDS IN A MIXTURE OF ACIDS USING CONDUCTIVITY METER

Short Procedure

Burette solution   :   Sodium Hydroxide

Pipette solution   :   20 ml of mixture of acids (HCl+CH₃COOH) + 30 ml distilled water

Place the conductivity cell in the beaker solution and conductance noted

A graph is plotted between volume of NaOH in x axis and conductance in y axis

The conductance decreases and gradually increases and suddenly increases.

From the graph volume of NaOH is obtained.
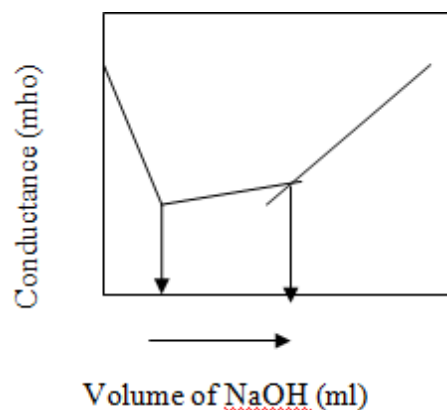
Equivalent weight of HCl $= 36.45$

Equivalent weight of $CH_3COOH = 63$

The amount of HCl= Strength of HCl x Equivalent weight of HCl

The amount of CH_3COOH = Strength ofCH_3COOH x Equivalent weight ofCH_3COOH

Model Graph

| Expt.No:07	**DETERMINATION OF STRENGTH OF ACIDS IN A MIXTURE**
Date:	**OF ACIDS USING CONDUCTIVITY METER**

AIM

To determine the amount of strong acid and weak acid (HCl&CH_3COOH) present in one litre of the given mixture of acid solution by conductometric titration using standard NaOH of strength N.

PRINCIPLE

Solutions of electrolytes conduct electricity due to the presence of ions. Since specific conductance of solution is proportional to the concentration of ions in it, conductance of the solution is measured during titration.

When a mixture of acids is titrated with a strong base, the acids are neutralized one by one, strong acid (HCl) first and weak acid (CH_3COOH) next. The neutralization reactions can be represented as,

$$HCl + NaOH \rightarrow NaCl + H_2O(I)$$

$$CH_3COOH + NaOH \rightarrow CH_3COONa + H_2O(II)$$
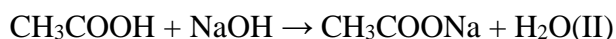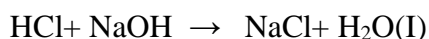
Since the fast-moving H^+ ions are replaced by slow moving Na^+ ions, decrease in conductance take place until the first end point (I) is reached. Since CH_3COONa is a strong electrolyte than CH_3COOH, conductivity slowly increases until all CH_3COOH is completely neutralized. When the second end point (II) is reached, addition of NaOH will cause sudden increase in the conductance. This is due to the presence of fast moving OH^- ions.

MATERIALS REQUIRED

Conductivity meter, Conductivity cell, Beaker, Standard NaOH solution, Given mixture of acids (HCl + CH_3COOH), Burette, Pipette.

Titration of mixture NaOH V_S (HCl + CH₃COOH)

S.No	Volume of NaOH added (ml)	Conductance (mho)

PROCEDURE:

The burette is cleaned with distilled water and filled with standard sodium hydroxide solution upto the mark. 20 ml of the given mixture of acids (HCl + CH$_3$COOH) is pipetted out into a clean 100 ml beaker and 30 ml of distilled water is added. The conductivity cell is placed in it, such that the electrodes are well immersed in the solution. The cell is connected to the conductivity meter.

The conductance is noted from the bridge for the mixture of acid alone (without the addition of sodium hydroxide). Now 1 ml of NaOH from the burette is added to the solution taken in the beaker, stirred for some time and the conductance is measured (the conductivity goes on decreasing upon the first end point). This process is repeated until at least five readings are taken after the first end point (I) has been reached.

After the end point, again NaOH is gradually added, which causes increase in conductance. This increase in conductance is observed until the second end point (II) is reached. After the second end point, a sudden increase in conductance is observed on further addition of NaOH. The readings are noted. Now a graph is plotted by taking volume of NaOH in the x-axis and conductance in the y–axis. The end point is noted. From the end point the amount of HCl and acetic acid present in 1 litre of the given mixture is calculated.

Calculation:

I) Calculation of strength of HCl

Volume of HClin the mixture	V_1	= 20 ml
Strength of HCl in the mixture	N_1	=
Volume of NaOH	V_2	= ------ ml (I titre value)
Strength of NaOH	N_2	= N

According to the law of volumetric analysis $V_1N_1 = V_2N_2$

$$N_2 = \frac{V_1N_1}{V_2}$$

Strength of HCl   =--------- N.

II) Calculation of amount of HCl

The amount of HCl present in   = Strength HCl x Equivalent weight of HCl

1000 ml of the given solution

=------------- x36.45

=...............gms.

III) Calculation of strength of CH₃COOH

Volume of CH₃COOH in the mixture	V_1 = 20 ml	
Strength of CH₃COOH in the mixture	N_1 =	
Volume of NaOH	V_2 = ------- ml (II – I value)	
Strength of NaOH	N_2=............N	

According to the law of volumetric analysis, $V_1N_1 = V_2N_2$

$$N_2 = \frac{V_1N_1}{V_2}$$

Strength of CH₃COOH   = ----------- N.

IV) Calculation of amount of CH₃COOH

The amount of CH₃COOH present   = Strength of CH₃COOH x

in 1000 ml of the given solution   Equivalent weight of CH₃COOH

=----------- x60

=.............gms.

RESULT

i) The amount of HCl present in 1000 ml of the given solution   =--------- gms.

ii) The amount of CH$_3$COOH present in 1000 ml of the given solution   =--------- gms.

DETERMINATION OF DISSOLVED OXYGEN CONTENT OF WATER SAMPLE BY WINKLER'S METHOD

Short Procedure:

Titration- I: Standardization of Sodium thiosulphate

Burette Solution   **: Sodium thiosulphate**

Pipette Solution   **: Potassium dichromate**

Reagents Added   **: 5 ml of dil.H_2SO_4 + 5 ml of 10%KI**

Indicator   **: 1 ml of Starch**

End Point   **: Colour change from blue to light green**

Potassium dichromate Vs Sodium thiosulphate

S.No	Volume of Potassium dichromate (ml)	Burette Reading (ml)		Volume of Sodium thiosulphate (ml)
		Initial	Final	
1.	20			
2.	20			

Concordant Value: ml

Calculation:

Volume of potassium dichromate $V_1 = 20$ ml

Strength of potassium dichromate $N_1 = \text{-------} N$

Volume of sodium thiosulphate $V_2 = \text{-------} $ ml

Strength of sodium thiosulphate $N_2 = \text{--------} N$

According to the law of Volumetric analysis, $V_1N_1 = V_2N_2$

$$N_2 = \frac{V_1N_1}{V_2}$$

Strength of Sodium thiosulphate   =   N

Expt.No:08	**DETERMINATION OF DISSOLVED OXYGEN CONTENT OF**
Date:	**WATER SAMPLE BY WINKLER'S METHOD**

AIM:

To determine the amount of dissolved oxygen in the given water sample by Winkler's method, a standard solution of $K_2Cr_2O_7$ of strength ---------is provided.
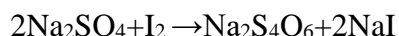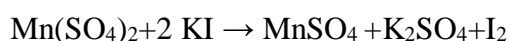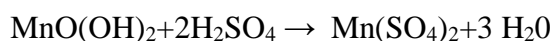
PRINCIPLE:

Oxygen is dissolved in water to the extent of 7-9 mgs/lit at temperature range of 25^0 $^-35^0$C. The estimation of dissolved oxygen in water is useful in studying the corrosion effects of boiler feed water and in studying water pollution. The dissolved oxygen content can be determined by Winkler's method (Potassium bromide + Potassium bromate). The water sample is collected carefully avoiding aeration/deaeration in ground stoppered iodine flask. Manganous sulphate solution in presence of alkali iodide is added to water in the iodine flask.

The following reactions occur

$$Mn^{2+} + 2OH^- \rightarrow Mn(OH)_2 \downarrow \text{ (White)}$$

$$Mn(OH)_2 + \frac{1}{2} O_2 \rightarrow MnO(OH)_2 \downarrow \text{(Yellow brown)}$$

The precipitate dissolves in Con H_2SO_4 liberating iodine and the liberated iodine is titrated against $Na_2S_2O_3$.

$$MnO(OH)_2 + 2H_2SO_4 \rightarrow Mn(SO_4)_2 + 3 H_2 0$$

$$Mn(SO_4)_2 + 2 KI \rightarrow MnSO_4 + K_2SO_4 + I_2$$

$$2Na_2SO_4 + I_2 \rightarrow Na_2S_4O_6 + 2NaI$$

MATERIALS REQUIRED

Sodium thiosulphate, Potassium dichromate solution, Dil H_2SO_4, 5% KI, Starch indicator, Manganese sulphate, Alkali iodide mixture, Con H_2SO_4, Burette, Pipette, Iodine flask.

Short Procedure:

<u>Titration II</u>: Determination of Dissolved oxygen

Burette Solution   : Sodium thiosulphate

Pipette Solution   : Water sample

Reagents Added   : 5ml of dil.H_2SO_4 + 5ml of 10%KI

 Indicator   : 1 ml Starch

End Point   : Disappearance of blue colour

Equivalent weight of oxygen: 8

<p align="center">Water sample Vs Sodium thiosulphate</p>

S. No	Volume of water sample (ml)	Burette Reading (ml)		Volume of Sodium thiosulphate (ml)
		Initial	Final	
1.	100			
2.	100			

<p align="right">Concordant Value: ml</p>

Calculation

Volume of sodium thiosulphate V_1 =-------ml

Strength of sodium thiosulphate N_1 =------ N

Volume of water sample V_2 =100 ml

 Strength of water sample N_2 =

According to law of volumetric analysis , $V_1N_1= V_2N_2$

$$N_2 = \frac{V_1N_1}{V_2}$$

Strength of Water Sample   =   N

Amount of DO in one litre of the water sample = Strength of water sample x Eq.Wt of O_2 x1000

=   x 8 x 1000mgs

=   mgs/lit

PROCEDURE
Titration I: Standardization of Sodium thiosulphate

The burette is washed with distilled water and rinsed with given sodium thiosulphate solution.20 ml of 0.01N potassium dichromate solution is pipette out into clean conical flask.

About 5 ml of dilute sulphuric acid and 5 ml of 10% potassium iodide solution are added. The liberated iodine is titrated at once against sodium thiosulphate solution taken in the burette. When the solution in the conical flask becomes straw yellow colour, 1ml of freshly prepared starch solution is added as indicator and the titration is continued. The end point is the disappearance of blue colour and the appearance of light green colour. Titre value is noted and titrations are repeated for concordant value.

TitrationII: Determination of Dissolved oxygen

100-150 ml of water sample is taken in iodine flask. 2 ml alkali iodide mixture and 2 ml of 5% manganeous sulphate solution are added and stoppered properly. The flask is inverted and shaken several times for thorough mixing of reagents. The flask is left aside for about 15 minutes for settling of theprecipitate. When half of the precipitate settles down, the stopper is removed and 2 ml of concentrated H_2SO_4isadded to get a clear yellow solution due to the liberation of iodine. 100 ml of this solution is measured and titratedagainst standardized sodium thiosulphate solution. When the solution becomes light yellow, 1 ml of starch indicator is added. The titration is continued until the blue colour disappears. Titre value is noted. From the titre value, the strength of dissolved oxygen is calculated and hence the amount of dissolved oxygen in the given water sample is calculated.

RESULT:

Amount of dissolved oxygen in water sample =_____mgs/ lit

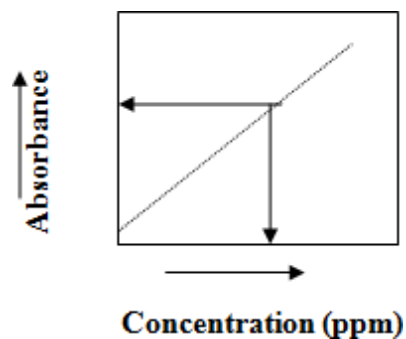# ESTIMATION OF IRON CONTENT OF WATER SAMPLE USING SPECTROPHOTOMETER

Short Procedure

- Spectrophotometer is calibrated to zero absorbance using distilled water.

- Absorbance is noted for iron solutions of 1, 2, 3, 4, 5, 6 ppm and unknown concentration.

- A graph is plotted between concentration in x axis and absorbance in y axis.

- The concentration of iron present in given water sample is determined from the graph.

Preparation of various concentrations of Fe^{3+} solution

S.No	Volume of stock solution (ml)	Volume of dil. HNO_3 (ml)	Volume of 1% NH_4SCN (ml)	Volume of distilled water (ml)	Concentration of iron (ppm)
1	10	5	10	75	1
2	20	5	10	65	2
3	30	5	10	55	3
4	40	5	10	45	4
5	50	5	10	35	5
6	60	5	10	25	6

Calibration curve (Absorbance Vs Concentration)

Expt.No:09	ESTIMATION OF IRON CONTENT OF WATER SAMPLE USING
Date:	SPECTROPHOTOMETER

AIM

To estimate the amount of iron present in given water sample by spectrophotometry using ammonium thiocyanate solution.

MATERIALS REQUIRED

Spectrophotometer, Ferrous ammonium sulphate solution, Ammonium thiocyanate solution, Nitric acid, Standard flasks, Graduated pipette, Distilled water.

PRINCIPLE

When a monochromatic light falls on a coloured homogeneous medium, a portion of the light is absorbed and a portion is reflected and the remaining is transmitted. The intensity of a transmitted light beam of monochromatic light decreases exponentially as the concentration of the absorbing substance in the solution increases and the path length of the light through the solution increases. This is known as Beer Lambert's law and can be written mathematically as,

$$\log I_o/I = \epsilon cx = A$$

Where I_o = intensity of incident light

I = intensity of transmitted light

ϵ = molar absorption coefficient

x = Thickness of the cell

c = concentration of the solution

A = absorbance

From the equation, it is seen that absorbance (A) is directly proportional to the concentration and thickness of the cell. Spectrophotometer is an instrument used to measure the intensity of the light absorbed by a substance. Solutions which are coloured or solutions which can be coloured by the addition of suitable reagent can be studied using this instrument.

In the determination of iron, ferrous ion (FAS) solution is acidified with nitric acid to oxidize Fe^{2+} into Fe^{3+}. Ferric ion does not give any colour to the solution. But it gives blood red colour when it reacts with ammonium thiocyanate forming a six coordinated, octahedral complex.

Measurement of absorbance:

Blank solution (Distilled water) = Zero absorbance, λ_{max} = 480 nm

S.No	Concentration		Absorbance
	ppm	**N**	
1	1	0.01 N	
2	2	0.02 N	
3	3	0.03 N	
4	4	0.04 N	
5	5	0.05 N	
6	6	0.06 N	
7	Sample		

Calculation

Amount of iron present in the given water sample = Concentration x Equivalent weight of iron x 1000

= ------- x 55.85x1000

=.................ppm.

$$Fe^{3+} + 6\ NH_4SCN \rightarrow [Fe(SCN)_6]^{3-} + 6NH_4^+$$
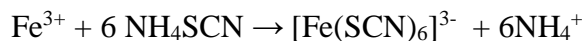
Blood red colour complex

This complex has maximum absorption at $\lambda= 480$ nm. To measure the intensity of incident light (I_ϕ) a blank solution using distilled water is used in the cell and the transmitted light is adjusted for cent percent transmission. A calibration curve is drawn by measuring the absorbance of solutions of known concentration. The absorbance of unknown solution is measured and the concentration of unknown solution is determined from the calibration curve.

PROCEDURE

I) Preparation of stock solution

A stock solution of ferric ion (0.1N) is prepared by dissolving 0.0838 gms of ferrous ammonium sulphate with 1ml of concentrated HNO_3 and made upto 1litre in a standard flask using distilled water.

$$\boxed{1\text{ml of stock solution} = 10 \text{ ppm of iron.}}$$

From this stock solution, various concentrations are prepared (1,2, 3, 4, 5 & 6 ppm).

II) Estimation of iron content

The spectrophotometer is switched on and warmed up for 10 minutes. The monochromator is adjusted for $\lambda=480$ nm. The blank solution (distilled water) is kept in the cell and the absorbance is measured for which the absorbance is zero and transmittance is100%. Now the absorbance of all the standard solutions is similarly measured. Also the absorbance of unknown solution is measured.

The calibration graph is drawn between the concentration and absorbance, from which the amount of iron present in the given water sample is measured.

RESULT

The amount of iron present in the given water sample = ----------ppm.

DETERMINATION OF MOLECULAR WEIGHT OF POLY VINYL ALCOHOL USING OSTWALD VISCOMETER

Short Procedure

- The flow time of solvent water is note.
- The flow time of different concentration of PVA are noted.
- A graph is plotted between concentration in x axis and η in y axis.
- A straight line is obtained. The intercept of the graph gives the intrinsic viscosity((η_i).

Molecular weight of the polymer

$$M = \text{Antilog} \left(\frac{\text{Log } \eta_i - \log \overline{K}}{a} \right)$$

Model Graph
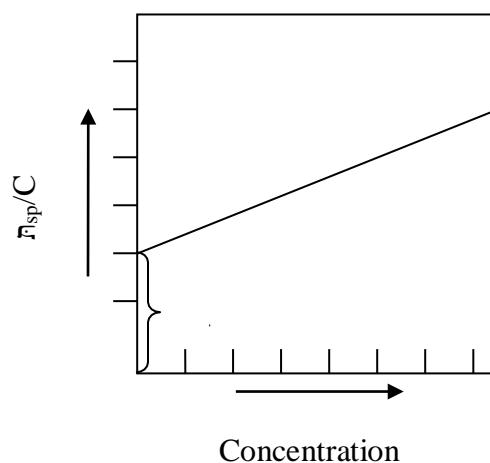
Concentration

S.No	Volume of 1% polymer solution (ml)	Volume of water (ml)	Concentration (C)
1	4	16	0.2
2	8	12	0.4
3	12	8	0.6
4	16	4	0.8
5	20	0	1

Expt.No:10	DETERMINATION OF MOLECULAR WEIGHT OF
Date:	POLYVINYL ALCOHOL USING OSTWALD VISCOMETER

AIM

To determine the molecular weight and degree of polymerization of polyvinyl alcohol using Oswald's viscometer, a 1% polymer solution is provided.

PRINCIPLE

Molecular weight of a polymer is nothing but the average molecular weight. This can be determined by measuring the intrinsic viscosity (η_i) of a dilute polymer solution. This intrinsic viscosity is related to the molecular weight by the following relationship.

$\eta_i = KM^a$ (Mark Hawniks equation)

where, η_i = Intrinsic viscosity, 'K' and 'a' are constants for a given polymer. The value of 'K' and 'a' for polyvinyl alcohol in water at 30^0C are K = 4.53 x 10^{-4} g / ml and a=0.64. M is the average molecular weight.

Degree of polymerization (D_p) provides another way of expressing the molecular weight as follows.

$M = D_p$ x m

where M = molecular weight of the polymer.

D_p = Degree of polymerization.

m = molecular weight of the monomer or the repeat unit.

MATERIALS REQUIRED

Ostwald viscometer, stop clock, 100 ml standard flasks, polyvinyl alcohol, distilled water.

Flow time of the pure solvent (t_O)=   sec

S.No	Concentration(C) %	Flow time t(sec)	Relative viscosity $\eta r = t/to$	Specific viscosity $\eta sp = t/to - 1$	Reduced viscosity $\eta red = \eta sp / C$
1	0.2%				
2	0.4%				
3	0.6%				
4	0.8%				

CALCULATION:

i)   Molecular weight of the polymer

Mark–Hawniks equation is **,**

$\eta_i = KM^a$

$\log \eta_i \quad = \log K + a \log M$

$\log M \quad = \underline{\log \eta_i - \log K}$

$\qquad\qquad\qquad a$

$M \quad = \text{Antilog} \left(\dfrac{\log \eta_i - \log K}{a} \right)$

$\quad = \underline{\quad\quad}$

$\quad = \underline{\dots\dots\dots\dots}gm.$

where $K = 4.53 \times 10^{-4}$ g /ml and a =0.64

PROCEDURE:

Preparation of polymer solutions of different concentrations

Polymer solutions of different concentration 0.25% ,0.30%, 0.45%, 0.60%, 0.75% are prepared from the given polymer stock solution.

Flow time of solvent:

15 ml of the solvent is taken into the viscometer and is sucked through the capillary tube upto the upper mark, without any air bubbles. Note the flow time of the solvent to flow from the upper mark to the lower mark (t_O).

Flow time of polymer solutions:

Fill the viscometer with 15 ml of one of the polymer solution into the viscometer and determine the flow time as before. Similarly, the flow time of all other polymer solution are determined from the flow times (η_{sp}/C) can be calculated. Graph is plotted between η_{sp}/C Vs concentration. A straight line is obtained with an intercept called intrinsic viscosity (η_i).

RESULT:

The molecular weight of the given polymer =

PREPARATION OF Na_2CO_3 AS A PRIMARY STANDARD AND ESTIMATION OF ACIDITY OF A WATER SAMPLE USING THE PRIMARY STANDARD

Calculation of Molarity of Na_2CO_3

Weight of Salt + bottle (W_1) =.................gm

Weight of empty bottle (W_2) = ... gm

Weight of salt   W $= (W_1 - W_2)$ =------ gm

The Molarity of Standard solution M_1 = [W/Mol.weight] x [1000/Volume of solution]

                                                 = [W/106] X [1000/100]

                                                 =

Expt.No:11	**PREPARATION OF Na₂CO₃ AS A PRIMARY STANDARD**
Date:	**AND ESTIMATION OF ACIDITY OF A WATER SAMPLE USING THE PRIMARY STANDARD**

AIM

a) To prepare Na_2CO_3 solution as a primary standard using anhydrous Na_2CO_3 crystal.

b) To estimate the acidity of given water sample using a standard Na_2CO_3 solution.

PRINCIPLE

Acidity is generally measured by titration with sodium carbonate to an accepted pH value. Hydrogen ions present, in a sample as a result of dissociation (or) hydrolysis of solutes, reacts with additions of standard alkali (Na_2CO_3). Acidity thus depends on end point of the indicator used. The colour change of phenolphthalein indicator is lose to pH at 25 ^{0}C corresponds to stoichiometric neutralization of carbonic acid to bicarbonate.

$$2H^+ + Na_2CO_3 \quad \rightarrow \quad H_2O + CO_2 + 2\ Na^+$$

MATERIALS REQUIRED

Burette, Measuring Jar, Conical flask, Weighing bottle, Std. Flask, Pipette, Burette, Electronic balance, Na_2CO_3, Methyl orange and Phenolphthalein.

PROCEDURE

(a) Preparation of standard Na₂CO₃ Solution

Take approximately 1.06 gm of the Na_2CO_3 salt in a weighing bottle and weigh it accurately by using digital balance. Now, transfer the salt into a 100 ml standard flask through funnel and dissolve the salt with minimum amount of distilled water. Then make-up the solution up to the mark of the standard flask and shake well to get uniform concentration. Later, the Molarity of Na_2CO_3 solution (M_1) can be calculated.

Short Procedure:

Titration I : Estimation of acidity of water

Burette Solution	: **Sodium Carbonate**
Pipette Solution	: **Water Sample**
Indicator	: **i) Phenolphthalein**
	ii) Methyl orange
End point	: **i) Disappearance of pink colour**
	ii) Colour change from yellow to red orange

Water sample Vs Standard Na_2CO_3

S.No	Volume of water sample (ml)	Burette Reading (ml)			Concordant Value (ml)	
		Initial	Methyl orange End point [M]	Phenolphthalein End point [P]	$[M]V_1$	$[P]V_2$
1.	20					
2.	20					

Calculation:

Mineral acidity

Volume of Na_2CO_3 V_1 (M)   =................... ml

Normality of Na_2CO_3 N_1 =................... N

Volume of water sample V_2 = ...ml

Strength of water sample (acidity) N_2 =

$$N_2 = \frac{V_1N_1}{V_2}$$

Acidity   =

Mineral acidity   =................N

Total acidity

Volume of Na_2CO_3 V_2 (P)   = ... ml

Normality of Na_2CO_3 N_1 = ... N

Volume of water sample V_2 =...............ml

Strength of water sample (acidity) N_2 =

Total acidity

Total acidity   = ------------ N

(b) Estimation of acidity of water

Burette is washed with distilled water and rinsed with standard Na_2CO_3 solution. Then the burette is filled with the same solution up to the zero mark without any air bubbles.

20 ml of water sample is pipetted out into a clean conical flask then few drops of methyl orange indicator are added to the conical flask. The solution is titrated against Na_2CO_3 solution till the colour changes from orange red to yellow. The volume consumed by water sample is noted. Then 2 to 3 drops of phenolphthalein indicator is added to the same solution. The titration is continued till the colour of water sample is turned to pale pink. The end point is noted. The titration is repeated until the two concordant values are obtained.

RESULT

Molarity of the Na_2CO_3 solution　　=

The given sample contains

1. Mineral acidity　　　　　=

2. Total acidity　　　　　　=

ESTIMATION OF TDS OF A WATER SAMPLE BY GRAVIMETRY

Short Procedure:

- An empty porcelain dish is washed and dried to 180 $^\circ$C.

- It is cooled and weight noted as w_1 gms.

- 50 ml of the filtered water sample is heated to 105°C, for drying. Let its weight be w_2 gms.

- The total dissolved solids (TDS) is calculated using the formula

$$\text{TDS} = \frac{\text{Weight of residue(mg)}}{\text{Volume of the sample (ml)}}$$

Tabulation

S. No	Description	Symbol	Weight(gms)
1.	Volume of the sample	v	
2,	Weight of the empty porcelain dish	w_1	
3.	'Weight of the dish + Residue	w_2	
4.	'Weight of the Residue	w_2-w_1	

Calculation

Weight of residue $w= w_2-w_1$

                                     = ... gms

                                     = ----------- x 1000 mgs

                                     = ... mgs.

Total Dissolved solids (mg/lit)

$$\text{TDS} = \frac{\text{Weight of residue(mg)}}{\text{Volume of the sample(ml)}}$$

                 = $\frac{\ \ \ \ \ \ \ \ }{\text{---------}}$

                 = ----------x 1000 mg/lit

                 = ... mg/lit

Expt.No:12	**ESTIMATION OF TDS OF A WATER SAMPLE BY GRAVIMETRY**
Date:	

AIM

To estimate the amount of TDS present in 100 ml of a given water sample by gravimetric method.

PRINCIPLE

A well-mixed water sample is filtered through a whattman filter paper. The filtrate is evaporated to dryness in a pre-weighed porcelain dish. The crucible is then dried to constant weight at 179 – 180 ^{o}C. The increase in crucible weight represents the total dissolved solids.

MATERIALS REQUIRED

Porcelain dish, Desiccator, Analytical balance, Hot air oven, Water bath, Whatman filter paper, 100 ml standard flask, 20 ml graduate pipette

PROCEDURE

An empty porcelain dish is washed and dried in a hot air oven at 180°C. It is then cooled in a desiccator and weighed in an analytical balance. Let its weight be $w_1$gms.

The given water sample is filtered through Whatman filter paper. 50 ml of the filtered water sample is taken in a porcelain dish and kept in an oven, maintained at 105°C, for drying. Drying is done for 1 to 2 hours till the porcelain dish (sample) attain the constant value.

The porcelain dish is cooled in a desiccator and weighed carefully. Let its weight be $w_2$gms.

The total dissolved solids (TDS) is calculated using the formula

$$TDS = \frac{Weight\ of\ residue(mg)}{Volume\ of\ the\ sample(ml)}$$

RESULT

Amount of Total Dissolved Solids (TDS) =---------- mgs/lit of a water sample

ESTIMATION OF IRON CONTENT OF THE GIVEN SOLUTION USING POTENTIOMETER

Short Procedure

Burette solution   : **Potassium dichromate**

Pipette solution   : **20 ml of ferrous ion solution + 10 ml of dil.H_2SO_4+20 ml distilled water**

The platinum electrode coupled with saturated calomel electrode is dipped in beaker solution and emf noted.

A graph is plotted between volume of $K_2Cr_2O_7$ in x axis and emf in y axis
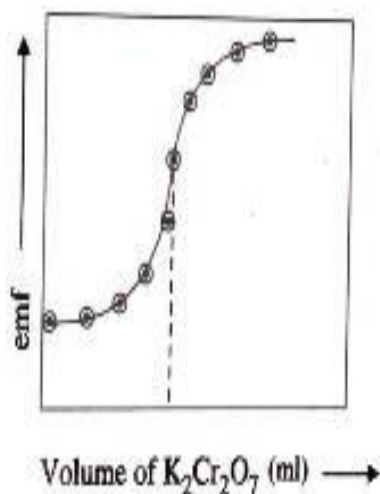
The potential gradually increases and suddenly increases near end point.

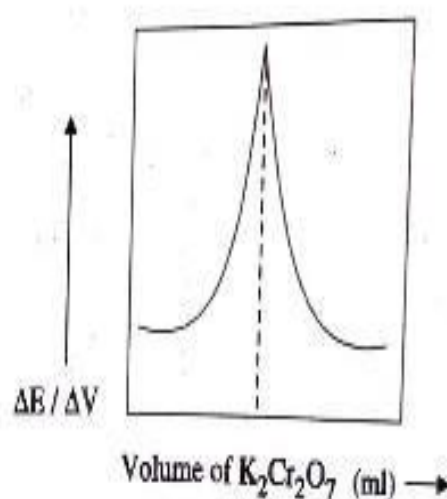From the graph volume of $K_2Cr_2O_7$ is obtained.

Equivalent weight of ferrous ion = 55.85

The amount of ferrous ion = Strength of ferrous ion x 55.85

Graph 1: Volume of $K_2Cr_2O_7$

Graph 2: Volume of $K_2Cr_2O_7$

Volume of $K_2Cr_2O_7$ (ml) ⟶

Volume of $K_2Cr_2O_7$ (ml) ⟶

Expt.No:13	ESTIMATION OF IRON CONTENT OF THE GIVEN SOLUTION
Date:	USING POTENTIOMETER

AIM

To estimate the amount of iron (ferrous ion (Fe^{2+}) present in 100 ml of the given solution using potentiometer. A standard solution of potassium dichromate of strength …… N is provided.

PRINCIPLE

Potentiometeric titrations depend on measurement of emf between reference electrode and an indicator electrode. When a solution of ferrous iron is titrated with a solution of potassium dichromate, the following redox reaction takes place.

$$6Fe^{2+} + Cr_2O_7^{2-} + 14H^+ \rightarrow 6Fe^{3+} + 2Cr^{3+} + 7H_2O$$
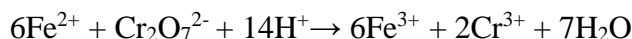
During the titration Fe^{2+} is converted into Fe^{3+} whose concentration increases. At the end point, there will be a sharp change due to sudden removal of all Fe^{2+} ions. The cell is set up by connecting this redox electrode with a calomel electrode as shown below.

$$Hg/Hg_2Cl_{2(s)}, KCI // Fe^{2+}, Fe^{3+}/Pt$$

A graph between emf measured against the volume of $K_2Cr_2O_7$ added is drawn and the end point is noted from the graph.

MATERIALS REQUIRED

Potentiometer, Pt electrode, Saturated Calomel electrode, Standard $K_2Cr_2O_7$ solution, Given ferrous ion solution, Dil. H_2SO_4, Distilled water, Burette and pipette

PROCEDURE

The given iron (ferrous ion) solution is transferred into 100ml Standard flask and made up to the zero-mark using distilled water. 20 ml of this made-up solution is pipetted out into a clean 100 ml beaker. About 10 ml of $H_2 SO_4$ and 20 ml of distilled water are added in it. A platinum electrode is dipped into the solution. This electrode is then coupled with a saturated calomel electrode and the cell is introduced into potentiometric circuit.

Tabular column

S.No	Volume of $K_2Cr_2O_7$ (ml)	Emf (Volts)	ΔE (Volts)	ΔV (ml)	ΔE/ ΔV (Volts /ml)

TITRATION – I

The burette is washed well with water and rinsed with the given $K_2Cr_2O_7$ solution. It is then filled with the same upto zero mark and titrated against the ferrous iron solution taken in the conical flask. $K_2Cr_2O_7$ solution is added in portions of 1ml and the emf of the cell is measured after each addition. The addition of $K_2Cr_2O_7$ is continued even after the end point and the range at which end point lies is found out by plotting volume of $K_2Cr_2O_7$ added against emf (graph 1)

TITRATION – II

Another titration is carried out by adding std. $K_2Cr_2O_7$ solution in portion of 0.1 ml near the end point and the emf of the cell is measured after each addition. The addition of $K_2Cr_2O_7$ is continued even after the end point for further 1 ml. The accurate end point is determined by plotting $\Delta E/\Delta V$ Vs Volume of $K_2Cr_2O_7$ added (graph 2). From the endpoint, strength of the ferrous ion solution and hence its amount can be calculated.

Calculation

Calculation of strength of ferrous iron solution

Volume of ferrous ion	$V_1 = 20$ ml
Strength of ferrous ion	$N_1 =$
Volume of $K_2Cr_2O_7$	$V_2 =$ ----- ml
Strength of $K_2Cr_2O_7$	$N_2 =$ ------- N
According to the law of volumetric analysis	$V_1N_1 = V_2N_2$

$$N_2 = \frac{V_1N_1}{V_2}$$

Strength of ferrous ion solution = ------- N

Calculation of amount of ferrous ion

The amount of iron (ferrous ion) present in 1000 ml
of the solution

= ----------- N x 55.85

= ----------- gms/Lit.

The amount of iron (ferrous ion) present in 100 ml
of the given solution

= ---------Nx 55.85x 100/1000gms.

= ----------- gms.

RESULT

The amount of iron (ferrous ion) present in 100 ml of the given solution   = ------------ gms.

ESTIMATION OF SODIUM AND POTASSIUM PRESENT IN WATER USING FLAME PHOTOMETER

Short Procedure

- The flame photometer is calibrated with distilled water.

- The absorbance of NaCl solutions of known conentrations(1,2 ,4,6,8 ppm) were noted and a calibration graph is plotted between flame photometer reading and concentration of K^+ ions.

- The concentration for unknown solution is noted and amount od sodium present in the water sample is determined from the calibration curve.
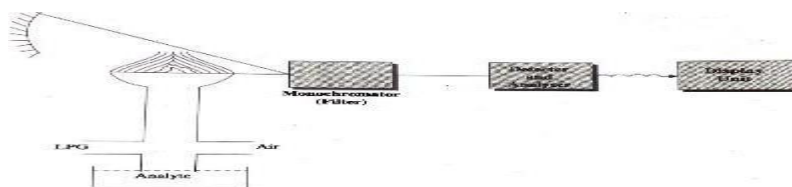

Table I Preparation of various concentration of Na^+ ion solution

SNo	Volume of stock solution (ml)	Volume of distilled water (ml)	Total volume (ml)	Concentration in ppm
1.	10	90	100	1
2.	20	80	100	2
3.	40	60	100	4
4.	60	40	100	6
5.	80	20	100	8

Table II Preparation of various concentration of K^+ ion solution

S.No	Volume of stock solution (ml)	Volume of distilled water (ml)	Total volume (ml)	Concentration in ppm
1.	10	90	100	1
2.	20	80	100	2
3.	40	60	100	4
4.	60	40	100	6
5.	80	20	100	8

Expt.No:14	ESTIMATION OF SODIUM AND POTASSIUM PRESENT IN
Date:	WATER USING FLAME PHOTOMETER

AIM

To estimate the amount of sodium and potassium present in the given water sample using flame photometer.

PRINCIPLE

Flame photometry for flame emission spectroscopy is based on the principle of emission of certain radiation in the visible region by a metal atom.

In this technique metal atom is thermally excited from the ground state. The excited atom emits light of particular intensity while returning to the ground state depending upon the concentration of metal ions. A water sample to be analysed is sprayed into the flame, where the water evaporate leaving the fine salt particles. This salt decomposes into constituent atoms, when they are heated to about 1700 $^{\circ}$C. The vapours containing metal atoms are excited by the thermal energy of the flame to higher energy state.

When the electrons fall down from the excited state to ground state, they emit radiation. The emitted radiation is measured and recorded.

The emission spectrum for each metal is different at its intensity depends on the concentration of the atoms in the flame.

Sodium produces a characteristic yellow emission at 589 nm.

Potassium produces a characteristic red emission at 766 nm.

Different standard solution (say 1, 2, 4,6 & 8 ppm) are prepared and the calibration curve is drawn between concentration (ppm) Vs intensity of emitted light (photometric readings).
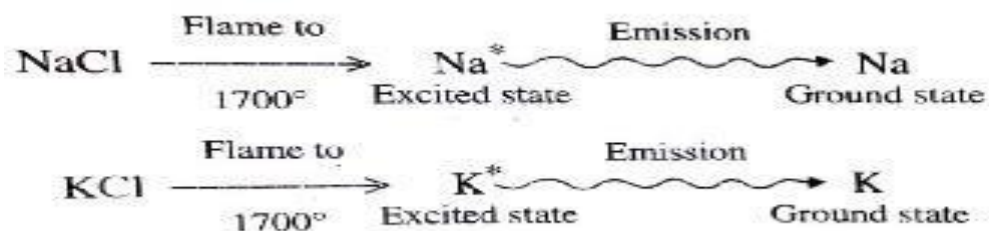
Materials Required

Flame photometer, Standard flask, NaCl solution, KCl solution, Distilled water.
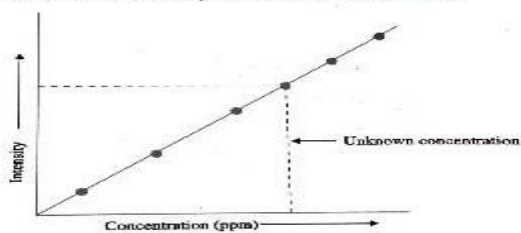
Graph: Calibration curve (Intensity Vs Concentration)

Concentration (ppm)

Intensity

Unknown concentration

TABLE - III

Determination of Na$^+$ ion in sample water

S.No	Concentration of NaCl	Intensity of emitted light (reading)
1.	1 ppm	
2.	2 ppm	
3.	4 ppm	
4.	6 ppm	
5.	8 ppm	
6.	Sample	

S.No	Concentration of KCl	Intensity of emitted light (reading)
1.	1 ppm	
2.	2 ppm	
3.	4 ppm	
4.	6 ppm	
5.	8 ppm	
6.	Sample	

Preparation of stock Na $^+$ ion solution

A stock solution of Na $^+$ ion is prepared by dissolving 25.4 gms of NaCl in 1 litre of distilled water.

1 ml of stock solution =10 ppm of Na$^+$ ion.

From the stock solution, various concentrations are prepared (1, 2, 4, 6 and 8 ppm) as shown in table II.

Preparation of stock K $^+$ ion

A stock solution of K $^+$ is prepared by dissolving 1.809 gms of KCl in 1 litre of distilled water.

1 ml of stock solution = 1 ppm of K $^+$ ion.

From the stock solution various concentration are prepared (1,2 ,4 6 and 8 ppm) as shown in the table II.

Working with instrument

The instrument is switched on. Air supply and gas supply are regulated. First distilled water is sent and ignition is started. After the instrument is warmed up for 10 minutes, the instrument is adjusted for zero reading in the display.

Determination of sodium in the water sample

NaCl solution of 8 ppm is sent and the reading is adjusted for100. Now the instrument is said to be calibrated. The solution of NaCl of other known concentration (1, 2, 4, 6 and 8 ppm) is sent one by one and the readings (intensity of emitted light) is noted. The calibration graph is drawn between concentration Vs intensity of emitted light (readings).

Now the sample is sent and the intensity of emitted light (reading) is noted. Then the concentration of sodium in the water sample is determined from the calibration curve.

Determination of potassium in the water sample

KCl solution of 8 ppm is sent and the reading is adjusted for 100. Now the instrument is said to be calibrated. The solution of KCl of other known concentration (1, 2, 4,6 and 8 ppm) is sent one by one and the reading is noted as before. The calibration graph is drawn between the concentration Vs intensity of emitted light (readings).

Now sample is sent and the reading is noted. Then the concentration of potassium in the water sample is determined from the calibration curve.

RESULTS

The amount of sodium present in the water sample =-------------gms.

The amount of potassium present in the water sample= ----------- gms.

PREPARATION OF NANOPARTICLES (ZnO) BY SOL-GEL METHOD

Short Procedure

Beaker solution : 100 ml of 1 M sodium hydroxide solution + 100 ml of the solution of 0.5 M zinc chloride solution+ 100 ml of a solution of 0.5 M zinc nitrate solution

Temperature and duration: 50º - 90º C and 50 minutes

Time of stirring : 2 hours

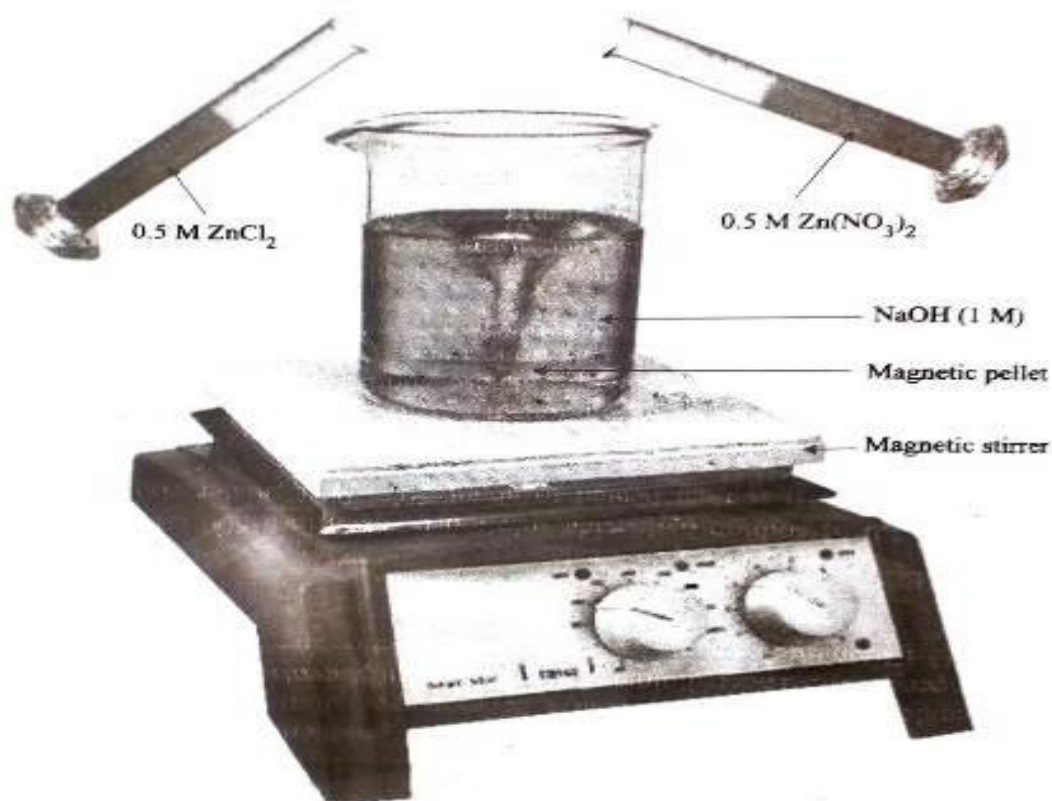**Product separation**: Precipitated ZnO is filtered, washed and dried.

Fig. : Preparation of ZnO nanoparticle by Sol-Gel process

Expt.No:15	**PREPARATION OF NANOPARTICLES (ZnO) BY SOL-GEL METHOD**
Date:	

AIM

Synthesis of ZnO nanoparticle using $ZnCl_2$, $Zn(NO_3)_2$ and NaOH as precursors.

MATERIALS REQUIRED

Zinc chloride, Zinc nitrate, Sodium hydroxide and Distilled water.

PRINCIPLE

The basic principle of sol-gel process involves production of solid material from small molecules. It involves conversion of monomers into a colloidal solution that acts as a precursor. The colloidal solution gradually evolves towards the formation of ZnO nanoparticles.

PROCEDURE

100 ml of 1 M sodium hydroxide solution is taken in 500 ml beaker and kept in a magnetic stirrer. It is then heated to about $50°$ and $90°$ C under constant stirring.

After obtaining the desired temperature, 100 ml of the solution of 0.5 M zinc chloride solution and 100 ml of a solution of 0.5 M zinc nitrate solution were slowly added (dripping) to the reaction mixture. These additions were done one by one for the period of 50 minutes. This process was done under constant stirring and the reaction temperature was maintained at desired value.

After dripping, stirring is continued for a period of two hours, maintaining the desired temperature. The ZnO precipitate formed in the reactor was filtered, washed and dried in a vacuum oven at $70°C$ for several hours and weighed.

RESULT

ZnO nanoparticles are prepared and the yield is =------ gms.

ESTIMATION OF NICKEL IN STEEL BY EDTA METHOD

Short Procedure

Titration- I: Standardization of EDTA

Burette Solution   : EDTA

Pipette Solution   : $ZnSO_4$

Reagents Added   : 10 ml of ammonia buffer

Indicator   : 2 drops of muroxide indicator

End Point   : Colour change from yellow to violet colour

$ZnSO_4$ Vs EDTA

S.No	Volume of $ZnSO_4$ (ml)	Burette Reading (ml)		Volume of EDTA (ml)
		Initial	**Final**	
1.	20			
2.	20			

Concordant Value: ml

Calculation

Volume of $ZnSO_4$ $V_1 = 20$ ml

Strength of $ZnSO_4$ $N_1 = \text{-------} N$

Volume of EDTA $V_2 = \text{-------}$ ml

Strength of EDTA $N_2 =$

According to the law of Volumetric analysis $V_1 N_1 = V_2 N_2$

$$N_2 = \frac{V_1 N_1}{V_2}$$

Strength of EDTA $N_2 = \text{-------} \quad N$

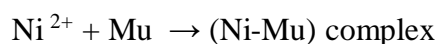| Expt.No:16	ESTIMATION OF NICKEL IN STEEL BY EDTA METHOD
Date:	

AIM

To estimate the amount of Nickel present in the given solution by EDTA method. A standard solution of zinc sulphate and EDTA as link solution are provided.

MATERIALS REQUIRED

EDTA, Fast sulphone black F and muroxide indicators, Ammonium chloride, Ammonium hydroxide, Pure and dry zinc sulphate and Nickel sulphate

PRINCIPLE

The nickel ions are determined using EDTA in presence of muroxide indicator

$$Ni^{2+} + Mu \rightarrow (Ni\text{-}Mu) \text{ complex}$$
$$\text{Yellow colour}$$
$$(Ni\text{-}Mu) \text{ complex} + EDTA \rightarrow Ni\text{-}EDTA + Muroxide$$
$$\text{Violet colour}$$

PROCEDURE

Titration I Standardization of EDTA

The Burette is washed with distilled water and then rinsed with a little amount of given EDTA solution. It is then filled with the same solution upto zero level without air bubbles. 20 ml of the given standard $ZnSO_4$ solution is pipetted out into a clean conical flask 10 ml of ammonia buffer solution and 2 drops of muroxide indicator are added. The solution turns yellow colour and it is then titrated against EDTA taken in the burette. The end point is the colour change from yellow colour to violet colour. The titration is repeated to get concordant values. From the volume of EDTA consumed, the strength of EDTA solution is calculate

Short Procedure

Titration- II Estimation of Nickel

Burette Solution : **EDTA**

Pipette Solution : **Nickel Solution**

Reagents Added : **10 ml of ammonia buffer**

Indicator : **2 drops of muroxide indicator**

End Point : **Colour change from yellow to violet colour**

EDTA Vs Nickel solution

S.No	Volume of nickel solution (ml)	Burette Reading (ml)		Volume of EDTA (ml)
		Initial	Final	
1.	20			
2.	20			

Concordant Value: ml

Calculation

Volume of EDTA $V_1 = 20$ ml

Strength of EDTA $N_1 = \ldots\ldots.N$

Volume of nickel solution $V_2 = \ldots\ldots.ml$

Strength of nickel solution $N_2 =$

According to the law of Volumetric analysis, $V_1N_1 = V_2N_2$

$$N_2 = \frac{V_1N_1}{V_2}$$

Strength of nickel solution $N_2 = \text{------ } N$

Calculation of amount of nickel

The amount of nickel present in whole of the given solution $= \text{------------}N \times 29.3$

$= \ldots\ldots\ldots\ldots\text{gms.}$

Titration II - Estimation of nickel

The given nickel solution is made up to 100 ml in a standard measuring flask. 20 ml of the solution is pipetted out into a clean conical flask. 10 ml of ammonia buffer solution and 2 drops of muroxide indicator was added. The solution turns yellow colour and it is then titrated against standardized EDTA taken in the burette. The colour changes from yellow to violet colour indicating the endpoint. The titration is repeated to get concordant values.

 Equivalent weight of Nickel =29.3

RESULT

Amount of Nickel present in the whole of the given solution=-------- gms.

PROXIMATE ANALYSIS OF COAL

Short procedure

- 1 gm of air-dried coal sample is heated in an electric oven at 110°C for one hour to determine the amount of moisture in coal.
- The coal sample is heated to 925°C for 7 minutes to estimate the volatile matter
- The residual coal is heated in a muffle furnace maintained of 725-750°C for ½ hour to determine the ash content.
- The sum total of percentage of moisture, volatile matter and ash, subtracted from 100, gives the percentage of fixed carbon in coal.

Calculation

i) Moisture Content

Weight of empty crucible $X_1 = $_____gms

Weight of crucible + sample $X_2 = $_____gms

Weight of crucible + sample after heating $X_3 = $----------gms.

% moisture $= (X_2 - X_3) / (X_2 - X_1) * 100$

                                                                                $= $................%

ii) Volatile Content

Weight of empty crucible $X_1 = $_____gms

Weight of crucible + sample $X_2 = $_____gms

Weight of crucible + sample (after the analysis of moisture) after heating $X_4 = $----------gms.

% volatile content $= (X_3 - X_4) / (X_2 - X_1) * 100$

                                                                                $= $................%

Expt.No:17	**PROXIMATE ANALYSIS OF COAL**
Date:	

AIM

To determine moisture, volatile and ash contents in a given coal sample by proximate analysis.

PRINCIPLE

Proximate analysis is type of analysis of coal. It informs about the practical utilization of coal. It is the determination of moisture, volatile matter, ash and fixed carbon. Calorific values mainly depend on the pressure of moisture, volatile matter and ash contents. So, it is essential to measure these items.

PROCEDURE

i) Moisture Content

The given coal sample is air dried. First empty silica crucible is exactly weighed in an analytical balance, let it wait be X_1 gms.

Now 1 gm of air-dried coal sample is taken in the silica crucible and let its wait be X_2 gms. The crucible is kept inside the electric oven, maintained at 110°C for one hour. After heating, the crucible is removed and kept in a desiccator for cooling and weighted. Let the weight of crucible after cooling be X_3 gms.

The difference in weight is reported in percentage as the amount of moisture in coal.

ii) Volatile Content

The coal sample, after the determination of moisture content, is heated in a furnace at 925°C for 7 minutes. After heating the crucible is removed from the furnace and cooled in a desiccator. After cooling, it is weighted. Let the weight of crucible be X_4 grams.

Loss in weight is reported as volatile matter on percentage basis.

iii) Ash Content

The residual coal, after the analysis of moisture and volatile content, is heated in a muffle furnace maintained of 725-750 ºC for ½ hour. Heating is done without crucible lid. The crucible is cooled first in air and then in desiccators. The crucible is then weighed, let the weight of the crucible be X_5 gms.

iii) Ash Content

Weight of empty crucible $X_1 =$ _____ gms

Weight of crucible + sample $X_2 =$ _____ gms

Weight of crucible + sample (after analysis of moisture + volatile content) after heating $= X_3 =$ ------- gms.

Ash Content $= (X_5 - X_1)/(X_2 - X_1) \times 100$

$= \ldots\ldots\ldots\ldots\%$

iv) Fixed Carbon Content

Fixed Carbon $= 100 - (\%\ \text{Moisture} + \%\ \text{Volatile matter} + \%\ \text{Ash})$

$= 100 - (\ldots.. + \ldots\ldots\ldots + \ldots\ldots)$

$= \ldots\ldots\ldots\ldots\%$

iv) Fixed Carbon Content

The sum total of percentage of moisture, volatile matter and ash, subtracted from 100, gives the percentage of fixed carbon.

RESULT

The coal sample contains

   i)   Moisture   =_____%

   ii) Volatile content   =_____%

   iii) Ash content   =_____%

   iv) Fixed carbon content   =_____%