Varuvan Vadivelan
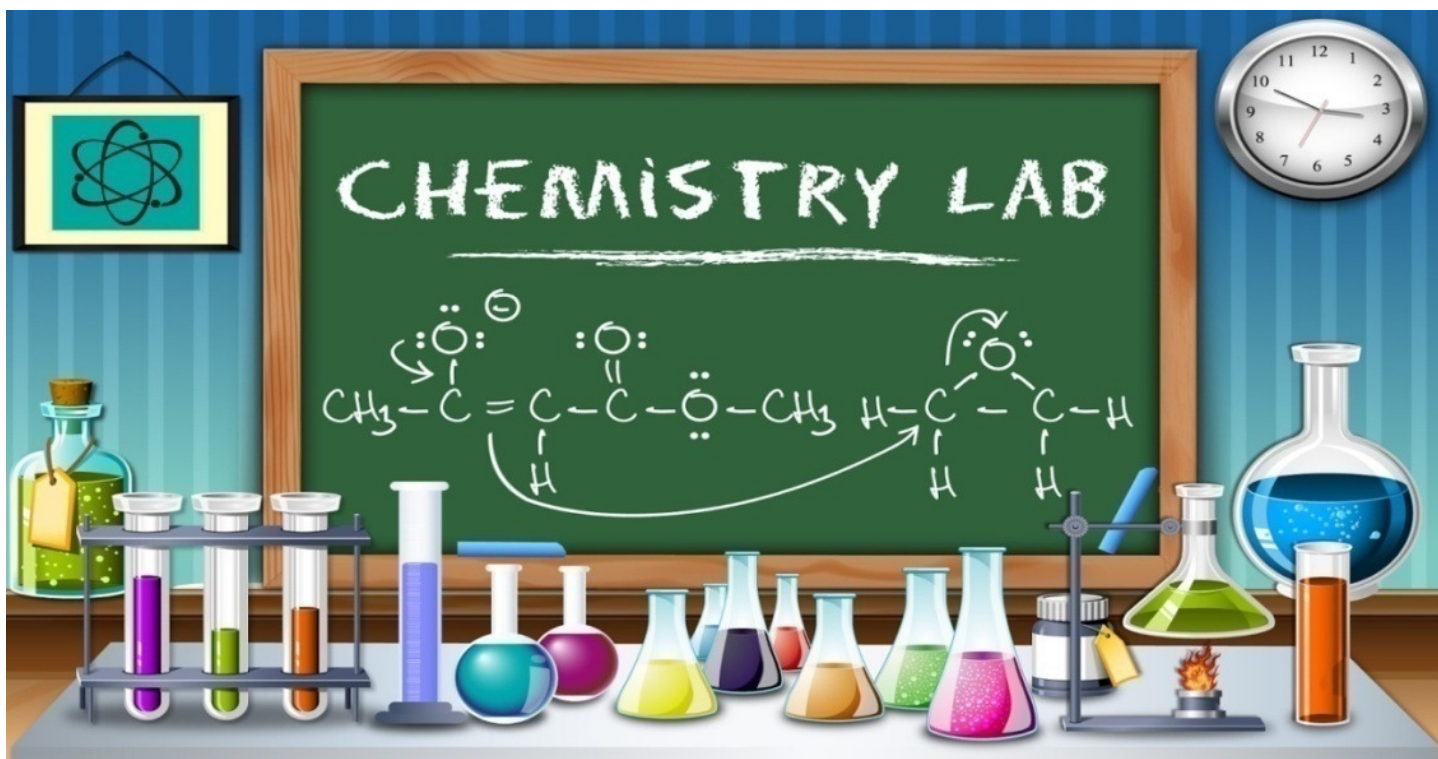## Institute of Technology
Dharmapuri – 636 703

LAB MANUAL

Regulation : 2013

Branch : *B.E.*–All Branches

Year & Semester : I Year / I Semester

GE6163-CHEMISTRY LABORATORY - I

ANNA UNIVERSITY: CHENNAI
SYLLABUS R – 2013
GE6163 - CHEMISTRY LABORATORY - I
LIST OF EXPERIMENTS

(Any 5 Experiments)

1. Determination of Dissolved Oxygen in Water [Winkler's Method].

2. Determination of Chloride Content in Water by Argentometric Method [Mohr's Method].

3. pH Metry - Determination of Strength of HCl by NaOH.

4. Determination of Strength of Acids in a Mixture Using Conductivity Meter.

5. Conductometric Titration of Strong Acid with Strong Base (Determination of NaOH).

6. Determination of Molecular Weight of Polyvinyl Alcohol Using Ostwald Viscometer.

7. Estimation of Iron Content of the Water by Spectrophotometry.

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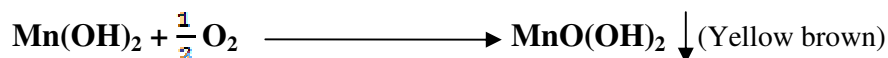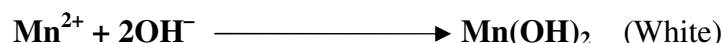| Ex. No.	Date	Name of Experiment	Signature of the Staff	Remarks
1.		Determination of Dissolved Oxygen in Water [Winkler's Method].		
2.		Determination of Chloride Content in Water by Argentometric Method [Mohr's Method].		
3.		pH Metry – Determination of Strength of HCl by NaOH.		
4.		Conductometric Titration of Strong Acid with Strong Base (Determination of NaOH).		
5.		Determination of Strength of Acids in a Mixture Using Conductivity Meter.		
6.		Determination of molecular weight of polyvinyl alcohol using Ostwald viscometer.		
7.		Estimation of iron content of the water sample using spectrophotometer.		

EX. NO: 1
DATE:

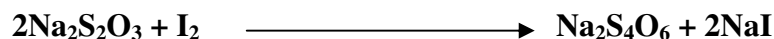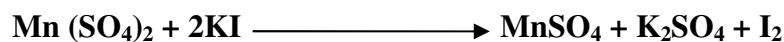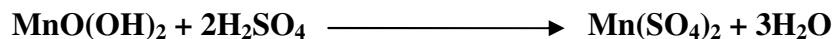# DETERMINATION OF DISSOLVED OXYGEN IN WATER
[Winkler's Method]

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 ……..N is given.

PRINCIPLE

Oxygen is dissolved in water to the extent of 7–9 mgs/lit at a temperature range of 25°C - 35°C. The estimation of dissolved oxygen in water is useful in studying corrosion effects of boiler feed water and in studying water pollution. The amount of dissolved oxygen in water is estimated using Winkler's reagent (Potassium bromide + Potassium bromate). Water sample is collected carefully avoiding aeration/deaeration in ground stoppered flask. Initially manganous sulphate and alkali-iodide reagents are added and the reactions occur as follows.

$$Mn^{2+} + 2OH^- \longrightarrow Mn(OH)_2 \quad \text{(White)}$$

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

The precipitate dissolves in concentrated sulphuric acid liberating iodine and the liberated iodine is titrated against $Na_2S_2O_3$.

$$MnO(OH)_2 + 2H_2SO_4 \longrightarrow Mn(SO_4)_2 + 3H_2O$$

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

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

MATERIALS REQUIRED

1. Sodium thiosulphate solution, 2. Std. Potassium dichromate solution, 3. Dil. H_2SO_4, 4. 5% KI, 6. Starch indicator, 7. Manganese sulphate, 8. Alkali-iodide mixture, 9. Conc. H_2SO_4 10. Burette, pipette, iodine flask.

PROCEDURE

TITRATION – I
Standardisation of Sodium Thiosulphate

The burette is washed and rinsed with sodium thiosulphate solution. Then the burette is filled with the given sodium thiosulphate solution upto zero mark. 20 ml of ……..N potassium dichromate solution is pipetted out into a clean conical flask. To this, 5 ml of sulphuric acid and 15 ml of 5% potassium iodide solution are added. This is titrated against sodium thiosulphate solution. When the solution becomes straw yellow colour, starch indicator is added and then titration is continued. The end point is disappearance of blue colour and appearance of light green colour. The titration is repeated to get concordant value.

TITRATION – II
Determination of dissolved oxygen

100-150 ml of the water sample is taken in the iodine flask, 2ml of manganese sulphate and 2ml of alkali-iodide mixture are added. The stopper is replaced and the flask is inverted and shaked several times for the rough mixing of reagents. The flask is left aside for some time. When half of the precipitate settles down, the stopper is removed and 2ml of concentrated sulphuric acid is added.

The stopper is replaced and the flask is inverted several times for complete dissolution of the precipitate to get a clear yellow solution. 20 ml of this solution is pipetted out in a clean conical flask and titrated against standardized sodium thiosulphate solution. When the solution becomes light yellow starch indicator is added. The titration is continued until the blue colour disappears. From the titre value the strength of dissolved oxygen is calculated and hence the amount of dissolved oxygen in the water sample is calculated.

TITRATION – I

Potassium dichromate Vs. Sodium thiosulphate

Sl. No	Volume of Potassium dichromate ml	Burette Readings		Concordant Volume of Sodium thiosulphate ml	Indicator
		Initial ml	Final ml		
1.	20	0			Starch
2.	20	0			

CALCULATION:

Volume of **Potassium dichromate** $V_1 = 20$ ml

Strength of **Potassium dichromate** $N_1 = $ ………………N

Volume of **Sodium thiosuphate** $V_2 = $ ………………ml

Strength of **Sodium thiosuphate** $N_2 = ?$

According to the law of volumetric analysis

$$V_1N_1 = V_2N_2$$

$$N_2 = \frac{V1 \times N1}{V2}$$

$$N_2 = \frac{20 \times ……………… N}{……………}$$

Strength of Sodium thiosulphate =……………….N

TITRATION II

Water sample Vs. Sodium thiosulphate

Sl. No	Volume of Water Sample ml	Burette Readings		Concordant Volume of Sodium thiosulphate ml	Indicator
		Initial ml	Final ml		
1.	20	0			Starch
2.	20	0			

CALCULATION:

Volume of **Sodium thiosuphate** V_1 = ………………ml

Strength of **Sodium thiosuphate** N_1 = ………………N

Volume of **Water sample** V_2 = **20 ml**

Strength of **Water sample** N_2 =?

According to the law of volumetric analysis

$$V_1N_1 = V_2N_2$$

$$N_2 = \frac{V1 \times N1}{V2}$$

$$N_2 = \frac{20 \times ………………N}{………………}$$

Strength of Water sample =……………….N

Amount of dissolved oxygen in 1 litre of tap water = **Normality x Eq.wt. of O_2 x 1000 mgs**

= ……………….N x 8 x 1000

= ………………….**mgs/lit**

RESULT

Amount of dissolved oxygen in water sample = ……………….**mgs/lit.**

EX. NO: 2
DATE:

DETERMINATION OF CHLORIDE CONTENT IN WATER BY ARGENTOMETRIC METHOD [Mohr's Method].

AIM

To determine the amount of chloride present in 100 ml of the given water sample, being supplied with standard solution of sodium chloride of strength …………N and an approximately N/20 solution of silver nitrate.

PRINCIPLE

Generally water contains chloride ions (Cl^-) in the form of NaCl, KCl, $CaCl_2$ and $MgCl_2$. The concentration of chloride ion in water, more than 250 ppm, is not desirable for drinking purpose. The total chloride ion can be determined by argentometric method (Mohr's Method).

In this method Cl^- ion solution is directly titrated against $AgNO_3$ using potassium chromate (K_2CrO_4) as an indicator.

$$AgNO_3 + NaCl \longrightarrow AgCl \downarrow + NaNO_3$$

(in water)   (White precipitate)
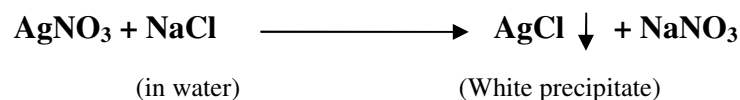
At the end point, when all the Cl^- ions are removed. The yellow colour of chromate changes into reddish brown due to the following reaction.

$$2AgNO_3 + K_2CrO_4 \longrightarrow Ag_2CrO_4 \downarrow + 2KNO_3$$

(Yellow)   (Reddish brown precipitate)
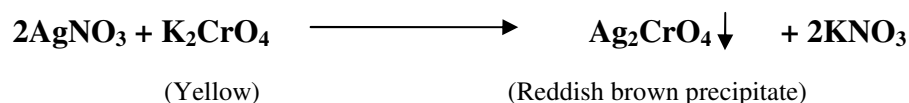
MATERIALS REQUIRED

1. Std. NaCl solution, 2. $AgNO_3$ solution, 3. 2% K_2CrO_4 indicator, 4. Burette, 5. Pipette, 6. Conical flask.

PROCEDURE

Step I

TITRATION – I
Standardisation of Silver nitrate

The burette is washed well with distilled water and rinsed with the small amount of $AgNO_3$ solution. It is then filled with the same solution upto the zero mark without any air bubbles.

The pipette is washed well with distilled water and rinsed with the small amount of standard NaCl solution. 20 ml of this solution is pipetted out into a clean conical flask. 1ml of 2% K_2CrO_4 indicator is added and titrated against $AgNO_3$ solution taken in the burette. The end point is the change of colour from yellow to reddish brown. The titration is repeated for concordant values.

Step II

TITRATION – II
Determination of chloride ions

20 ml of the given water sample is pipetted out into a clean conical flask and 1ml of 2% K_2CrO_4 indicator is added. It is then titrated against standardized $AgNO_3$ solution taken in the burette. The end point is the change of colour from yellow to reddish brown. The titration is repeated for concordant values.

Step I: STANDARDISATION OF SILVER NITRATE

TITRATION – I
Standard NaCl Vs. AgNO$_3$

Sl. No	Volume of Nacl ml	Burette Readings		Concordant volume of AgNO$_3$ ml	Indicator
		Initial ml	Final ml		
1.	20	0			K$_2$CrO$_4$
2.	20	0			

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

$$V_1 N_1 = V_2 N_2$$

$$N_2 = \frac{V1 \times N1}{V2}$$

$$N_2 = \frac{20 \times …………………N}{……………}$$

∴ Strength of Silver Nitrate = ………………N

Step II: DETERMINATION OF CHLORIDE ION

TITRATION – II
Water sample Vs. Std. AgNO₃

Sl. No	Volume of water sample ml	Burette Readings		Concordant volume of AgNO₃ ml	Indicator
		Initial ml	Final ml		
1.	20	0			K₂CrO₄
2.	20	0			

Calculation of Normality of the water sample (Chloride ion)

Volume of **water sample** $V_1 = 20$ ml

Strength of **water sample** $N_1 = ?$

Volume of **Silver Nitrate** $V_2 = $ …………..ml

Strength of **Silver Nitrate** $N_2 = $ ……………N

According to the law of volumetric analysis

$$V_1N_1 = V_2N_2$$

$$N_1 = \frac{V2 \times N2}{V1}$$

$$N_2 = \frac{…………… x …………………N}{20}$$

Strength of water sample = ……………….N

Calculation of amount of the chloride

Amount of the chloride ion present in
1 litre of the water sample

= **Equivalent weight of chloride ion X
Strength of the chloride ion**

= **35.46 x** ……………..**N**

= ………………**gms**

· Amount of the chloride ion present in100 ml of the given water sample

= ………………**gms x 100/1000**

= ………………**gms**

RESULT

1. Amount of chloride ion present in the whole of the given water sample = ………….**gms/mgs/ppm**

2. Amount of chloride ion present in the 100 ml of the given water sample = ………....**gms/mgs/ppm**

EX. NO: 3
DATE:

# pH METRY - DETERMINATION OF STRNGTH OF HCl BY NaOH

AIM

To determine the strength of given HCl by pH metry, a standard solution of NaOH of …………N is provided.

PRINCIPLE

Since the pH of the solution is related to the H$^+$ ion concentration by the following 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.

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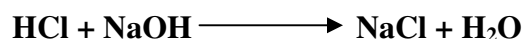
The increase in pH takes place until all the H$^+$ ions are completely neutralized (upto the end point). After the end point, further addition of NaOH increases the pH sharply as there is an excess of fast moving OH$^-$ ions.

MATERIALS REQUIRED

1. pH meter, 2. Glass electrode, 3. 100 ml Beaker, 4. Standard NaOH, 5. Given HCl, 6. Burette, pipette, glass rod etc., 7. Distilled water.

PROCEDURE

TITRATION – I

The given hydrochloric acid solution is transferred into 100 ml 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 and diluted by adding 20 ml of distilled water. The glass electrode is dipped in it and connected with a pH meter.

The burette is washed well with water and rinsed with a small amount of given NaOH solution. It is then filled with the same upto zero mark. Titration is carried out by adding std. NaOH solution in portions of 1ml from the burette to the HCl solution taken in the beaker and pH of the solution is noted for each addition. This process is continued until atleast 5 readings are taken after the end point, and the range at which the end point lies is found out by plotting volume of NaOH added against pH (graph I).

TITRATION – II

Another titration is carried out by adding std. NaOH solution in portions of 0.1 ml near the end point and pH of the solution is noted after each addition. The addition of NaOH is continued even after the end point for further 1 ml. The accurate end point is found out by plotting $\Delta pH/\Delta V$ against volume of NaOH added (graph II). From the end point, the strength of HCl solution and hence the amount of HCl is calculated.
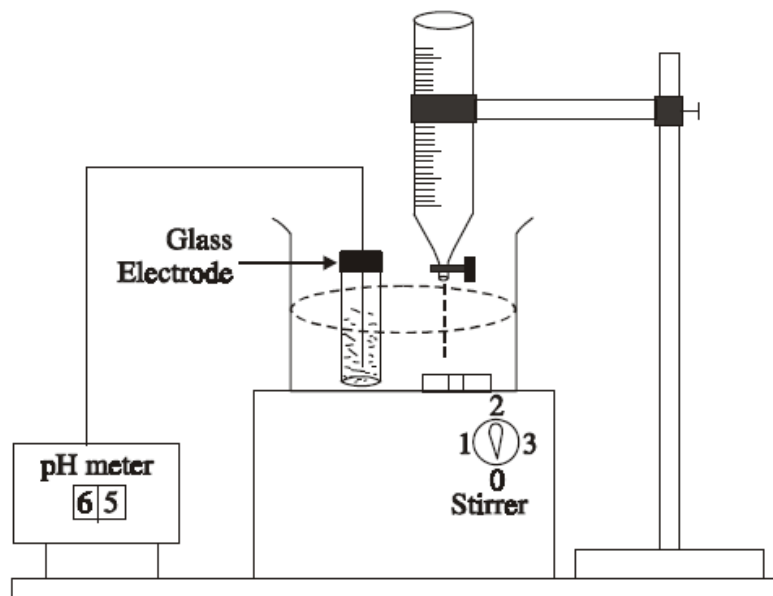
Determination of pH

TABLE – I (Titration – I)

Titration of HCl (20 ml) Vs. NaOH

S. No	Volume of NaOH ml	pH	Remarks
1.			
2.			
3.			Gradual increase
4.			
5.			
6.			
7.			End point
8.			
9.			
10.			Sudden increase
11.			
12.			
13.			

TABLE – II (Titration – II)

Titration of HCl (20 ml) Vs. NaOH

S. No	Volume of NaOH ml	pH	ΔpH	ΔV Ml	ΔpH / ΔV
1.					
2.					
3.					
4.					
5.					
6.					
7.					
8.					
9.					
10.					
11.					
12.					
13.					
14.					
15.					
16.					
17.					
18.					
19.					
20.					

Model Graph

Plot of pH Vs V

$$\frac{\Delta pH}{\Delta V} \text{ Vs V}$$
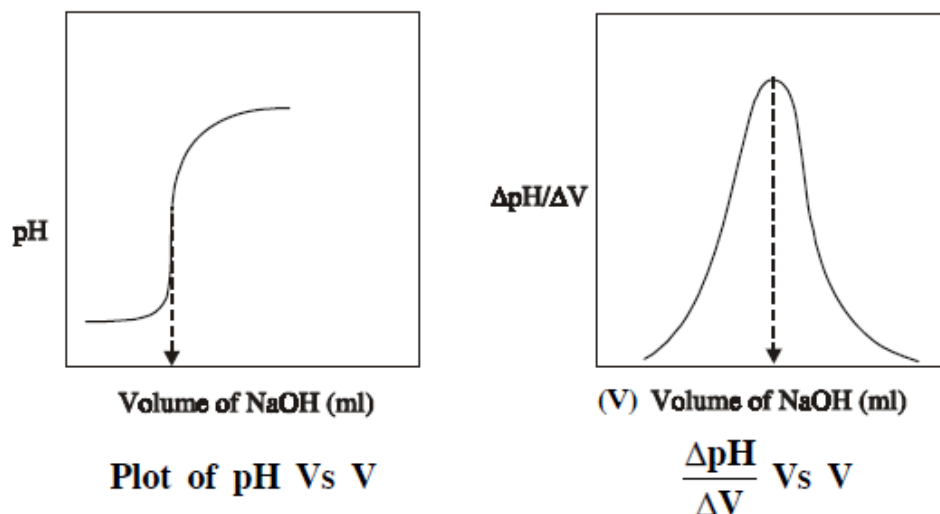
Calculation

Step I: Calculation of Strength of HCl

Volume of **HCl**	$V_1 = 20$ ml
Strength of **HCl**	$N_1 = ?$
Volume of **NaOH**	$V_2 = \ldots\ldots\ldots\ldots$ml (titre value)
Strength of **NaOH**	$N_2 = \ldots\ldots\ldots\ldots$N

According to the law of volumetric analysis,

$$V_1N_1 = V_2N_2$$

$$N_1 = \frac{V2 \times N2}{V1}$$

$$= \frac{\ldots\ldots\ldots X \ldots\ldots\ldots N}{20}$$

Strength of HCl $= \ldots\ldots\ldots\ldots$.N

Step – II: Calculation of amount of HCl

The amount of HCl present in 1000 ml of the given solution = …….N x **Eq. wt. of HCl (36.45)**

$$= \ldots\ldots\ldots\ldots \text{ x } 36.45$$

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

RESULT

1. Strength of the given HCl solution = ………………….**N.**

2. Amount of HCl present in 1 litre of the solution = ………………….**gms.**

EX. NO: 4
DATE:

CONDUCTOMETRIC TITRATION OF STRONG ACID WITH STRONG BASE (Determination of NaOH)

AIM

To determine the amount of sodium hydroxide present in 250 ml of the given solution by conductometric titration. You are provided with standard hydrochloric acid of ……………….N.

PROCEDURE

Solution of electrolytes conducts electricity due to the presence of ions. The specific conductance of a solution is proportional to the concentration of ions in it. The reaction between HCl and NaOH may be represented as,

$$\text{HCl} + \text{NaOH} \longrightarrow \text{NaCl} + \text{H}_2\text{O}$$

When a solution of hydrochloric acid is titrated with NaOH, the fast moving hydrogen ions are progressively replaced by slow moving sodium ions. As a result conductance of the solution decreases. This decrease in conductance will take place until the end point is reached. Further addition of alkali raises the conductance sharply ad there is an excess of hydroxide ions.

A graph is drawn between volume of NaOH added and the conductance of solution. The exact end point is intersection of the two curves.

MATERIALS REQUIRED

1. Conductivity bridge, 2. Conductivity cell, 3. 100 ml beaker, 4. Standard HCl, 5. Given NaOH solution, 6. Distilled water, 7. Burette, Pipette, glass rod etc.

PROCEDURE

TITRATION

The burette is washed well with distilled water and rinsed with the given NaOH solution. It is then filled with same upto the zero level. 20 ml of the standard HCl is pipetted out into a clean 100 ml beaker.

The conductivity cell is placed in it and immersed in the solution. The two terminals of the cell are connected with a conductivity bridge.

Now 1 ml of NaOH from the burette is added to the solution, taken in the beaker, stirred for some time and then conductivity is measured. (The conductivity is going on decreasing up to the end point).This process is repeated until at least five readings are taken after the end point has been reached.

Now the graph is plotted by taking volume of NaOH in the X-axis and conductance in the Y-axis. The end point is the intersection of the two curves. From the volume of NaOH, the strength and hence the amount of NaOH present in 250 ml of the solution is calculated.

Step I

TABLE – I
Titration of Standard HCl Vs. NaOH
Volume of HCl taken = 20 ml

S. No	Volume of NaOH added ml		Conductance mho		Remarks
1.	0	21			
2.	1	22			
3.	2	23			
4.	3	24			Decreases
5.	4	25			
6.	5	26			
7.	6	27			
8.	7	28			
9.	8	29			
10.	9	30			End point
11.	10	31			
12.	11	32			
13.	12	33			
14.	13	34			Increases
15.	14	35			
16.	15	36			
17.	16	37			
18.	17	38			
19.	18	39			
20.	19	40			

Graph: Conduction Vs. Volume of NaOH
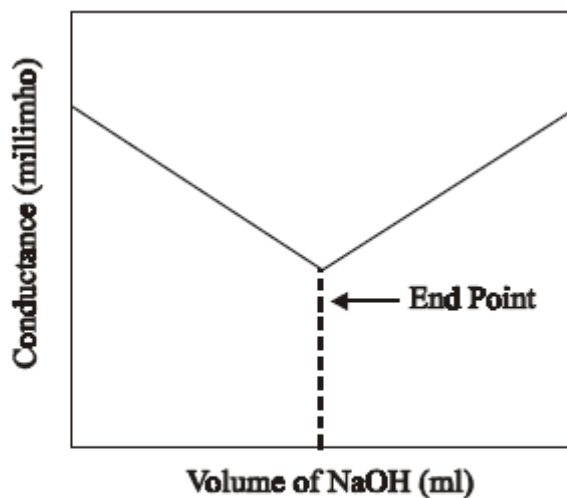
HCl Vs NaOH

Step II

Calculation of Strength of NaOH

Volume of **HCl**	**V$_1$ = 20 ml**
Strength of **HCl**	**N$_1$** = ……………..**N**
Volume of **NaOH**	**V$_2$** = ……………**ml** (titre value)
Strength of **NaOH**	**N$_2$ = ?**

According to the law of volumetric analysis,

$$V_1N_1 = V_2N_2$$

$$N_2 = \frac{V1 \times N1}{V2}$$

$$N_2 = \frac{20 \times \text{..................}}{\text{..............}}$$

Strength of NaOH = ………………..N

Calculation of amount of NaOH

The amount of NaOH present in 1000 ml of the given solution   **= 40 x** …………..**N**

= ………….gms

The amount of NaOH present in 250 ml of the given solution   **= 40 x** ………..**N x 250/1000**

= ………….gms

<u>**RESULT**</u>

1. The strength of given NaOH solution = ………………….**N**

2. The amount of NaOH present in 250 ml of the given solution = ………………..**gms.**

EX. NO: 5
DATE:

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

AIM

To determine the strength and amount of a strong acid and a weak acid (HCl and CH_3COOH) present in one litre of the given solution by conductometric titration. You are provided with standard NaOH solution of ……………….N.

PRINCIPLE

Solution of electrolytes conducts 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.

When the sodium hydroxide is added slowly from the burette to the solution, HCl (strong acid) gets neutralized first. Since the fast moving H^+ ions are replaced by slow moving sodium ions, decrease in conductance take place until the end point is reached.

$$HCl \ + \ NaOH \longrightarrow NaCl \ + \ H_2O \ (\text{ I}^{st} \text{ neutralization})$$
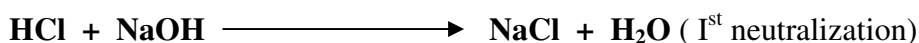
After the complete neutralization of all HCl, the neutralization of CH_3COOH starts,

$$CH_3COOH + NaOH \longrightarrow CH_3COONa + H_2O \ (\text{II}^{nd} \text{ neutralization})$$
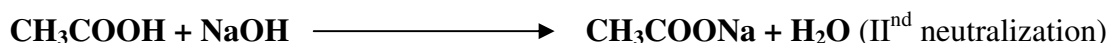
Since CH_3COONa is stronger electrolyte than CH_3COOH, conductivity slowly increases until all CH_3COOH is completely neutralized. When the end point is reached, addition of NaOH will cause sudden increase in the conductance. This is due to the presence of fast moving OH^- ions.

MATERIALS REQUIRED

1. Conductivity bridge, 2. Conductivity cell, 3. 100 ml beaker, 4. Standard NaOH, 5. Given HCl and CH_3COOH mixture, 6. Burette, Pipette, glass rod etc., 7. Distilled water.
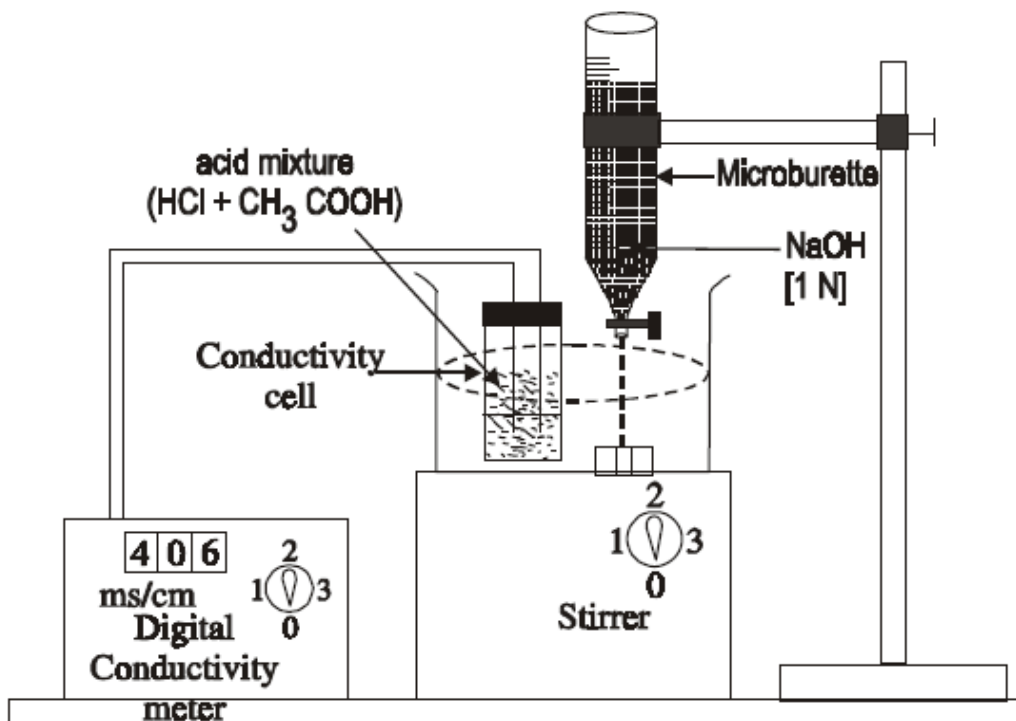
PROCEDURE

TITRATION

The given mixture of acids (HCl + CH₃COOH) is transferred into 100 ml standard flask and made upto zero mark using distilled water. 20 ml of the made up solution is pipetted out into a clean 100 ml beaker and the conductivity cell is placed in it and immersed in the solution. The two terminals of the cell are connected with a conductivity bridge.

The burette is washed well with water and rinsed with the given standard NaOH solution. It is then filled with NaOH solution up to the zero level.

Now 1 ml of NaOH from the burette is added to the solution, taken in the beaker, stirred for some time and then conductivity is measured. (The conductivity is going on decreasing up to the end point).This process is repeated until at least five readings are taken after the end point (A) 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 end point (B) is reached.

After the second end point, sudden increase in conductance is observed on further addition of NaOH. The readings are continuously measured for each addition of NaOH and are tabulated. Now the graph is plotted between the volume of NaOH Vs conductivity. From the graph the first end point (A) and the second end point (B) are noted. From the end points the strengths and amount of HCl and CH₃COOH present in 1 liter of the mixture of solution are calculated.
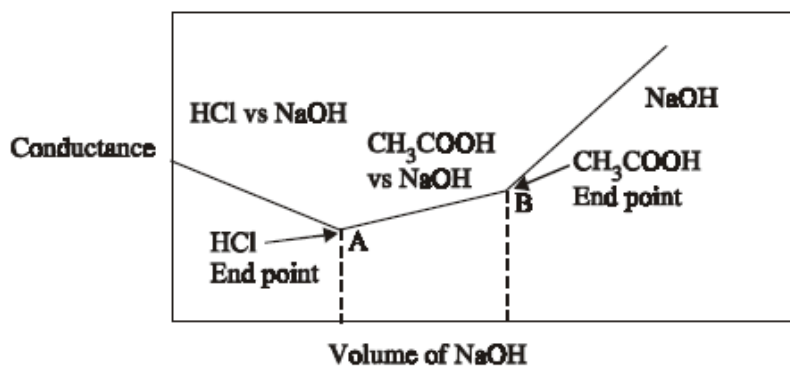
acid mixture
(HCl + CH₃ COOH)

Microburette

NaOH
[1 N]

Conductivity
cell

4 0 6
ms/cm
Digital
Conductivity
meter

2
1 ⃝ 3
0

2
1 ⃝ 3
0

Stirrer

Model graph

HCl vs NaOH

Conductance

CH₃COOH
vs NaOH

NaOH

CH₃COOH
End point

B

HCl
End point

A

Volume of NaOH

TABLE – I

Titration of mixture (HCl + CH₃COOH) Vs. NaOH

S. No	Volume of NaOH added ml		Conductance mho	
1.	0	20		
2.	1	21		
3.	2	22		
4.	3	23		
5.	4	24		
6.	5	25		
7.	6	26		
8.	7	27		
9.	8	28		
10.	9	29		
11.	10	30		
12.	11	31		
13.	12	32		
14.	13	33		
15.	14	34		
16.	15	35		
17.	16	36		
18.	17	37		
19.	18	38		
20.	19	39		

Step I

Calculation of Strength of HCl

Volume of the mixture **(HCl)**	V_1 = 20 ml	
Strength of the mixture **(HCl)**	N_1 = ?	
Volume of the **NaOH**	V_2 = …………… **(A) ml** (I^{st} titre value)	
Strength of the **NaOH**	N_2 = ………………N	

According to the law of volumetric analysis,

$$V_1N_1 = V_2N_2$$

$$N_1 = \frac{V2 \times N2}{V1}$$

$$= \frac{………ml \times …………N}{20}$$

Strength of HCl = ……………….N

Calculation of amount of HCl

∴ The amount of **HCl** present in 1000 ml of the given solution

= **Strength of HCl x Eq. wt. of HCl**

= ……………….N x **36.45**

= ……………….**gms**

Step I

Calculation of Strength of CH$_3$COOH

Volume of the mixture **(CH$_3$COOH)**	V_1 = 20 ml	
Strength of the mixture **(CH$_3$COOH)**	N_1 = ?	
Volume of the **NaOH**	V_2 = ………… **(B-A) ml** (IInd titre value)	
Strength of the **NaOH**	N_2 = ………………N	

According to the law of volumetric analysis,

$$V_1N_1 = V_2N_2$$

$$N_1 = \frac{V2 \times N2}{V1}$$

$$= \frac{\ldots\ldots ml \times \ldots\ldots N}{20}$$

Strength of CH_3COOH = ………………N

Calculation of amount of CH₃COOH

∴ The amount of CH_3COOH present in 1000 ml of the given solution = **Strength of CH₃COOH x Eq.wt. of CH₃COOH**

= ………………**N x 60**

= ………………**gms.**

RESULT

1. The strength of HCl present in 1 litre of the given solution   = …………………**N**

2. The amount of HCl present in 1 litre of the given solution   = ………………..**gms**

3. The strength of CH_3COOH present in 1 litre of the given solution   = …………………**N**

4. The amount of CH_3COOH present in 1 litre of the given solution   = ………………..**gms**

EX. NO: 6
DATE:

DETERMINATION OF MOLECULAR WEIGHT OF POLYVINYL ALCOHOL USING OSTWALD VISCOMETER

AIM

To determine the molecular weight of the polyvinyl alcohol using Ostwald's viscometer, a 1% PVA solution is provided.

PRINCIPLE

Molecular weight of PVA means average molecular weight of the polymer. This can be determined by measuring the intrinsic viscosity (η_i) of a dilute PVA solution. This intrinsic viscosity is related to the molecular weight by the following relationship.

$$\eta_{i=kM}{}^{a} \text{ (Mark Hownik equation)}$$

where, $\eta_i =$ Intrinsic viscosity

k & a = Constants for a given PVA-solvent combination at a given temperature.

M = Average molecular weight.

Moleular weight constants (k and a) for some polymer / solvent systems are given in table III.

MATERIALS REQUIRED

1. PVA, 2. Distilled water, 3. Viscometer, 4. 50 ml std. flask , 5. 20 ml graduated pipette, 6. Stop watch.

Important Viscosity definitions

1. Relative viscosity (η_r) $\eta_r = \dfrac{\eta}{\eta_0} = \dfrac{t}{t_0}$

2. Specific viscosity (η_{sp}) $\eta_{sp} = \dfrac{\eta}{\eta_0} - 1 = \dfrac{t}{t_0} - 1$

3. Reduced viscosity (η_{red}) $\eta_{red} = \dfrac{\eta_{sp}}{c}$

4. Intrinsic viscosity (η_i) $\eta_{i=} \underset{C \xrightarrow{\text{lim}} 0}{} \dfrac{\eta sp}{C}$

Where,

η_i = Absolute viscosity of a PVA solution.

η_o = Absolute viscosity of a pure solvent

t = Flow time for the PVA solution

t_o = Flow time for the solvent (H_2O).

PROCEDURE

Step I: Preparation of PVA solutuions of different concentrations

PVA solutions of different concentrations, say 0.2%, 0.4%, 0.6%, 0.8% and 1% are prepared from the given polymer stock solution as in Table - I.

Step II: Determination of flow time of water

20 ml of the water is taken into the viscometer and is sucked through the capillary tube upto the uppermark, without any air bubbles. Now the flow time is noted for the water to flow from the uppermark (A_1) to lowermark (A_2).

Step 3: Determination of flow time of PVA solutions

Now fill the viscometer with 20 ml of one of the PVA solution (say I) into the viscometer and flow time is determined as before. Similarly the flow time of the other PVA solutions (IInd, IIIrd etc.,) are determined.

Note: For each PVA solution, the viscometer is washed and rinsed with water.

From the flow times, reduced viscosity (η sp/C) can be calculated. Graph is plotted between η sp/C vs concentration, straight line is obtained with an intercept is called intrinsic viscosity (η_i).

Model Graph
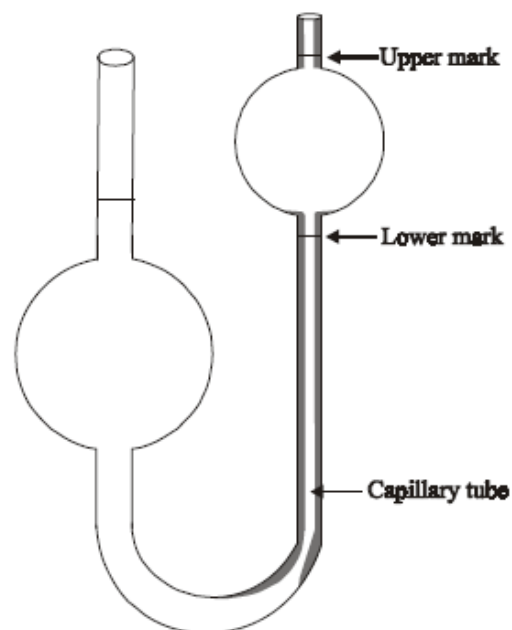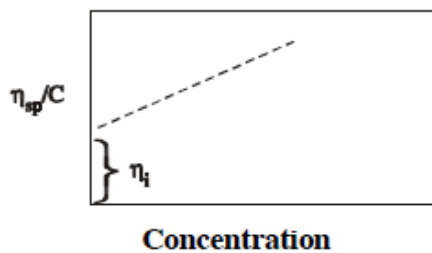
Ostwald Viscometer

TABLE - I

Preparation of various PVA solutions

S. No	Volume of 1% PVA solutions (Stock Solutions) ml	Volume of water ml	Concentration %
I	4	16	0.2
II	8	12	0.4
III	12	8	0.6
IV	16	4	0.8
V	20	0	1.0

<u>**TABLE – II**</u>

Viscosity data for PVA / water

Flow time of the pure (t_0) = ……. sec

S. No	Concentration %	Flow time t Sec	$\eta/\eta_0 = t/t_0 = \eta_r$	$t/t_0 - 1 = \eta_{sp}$	$\eta_{sp}/C = \eta_{red}$
I	0.2				
II	0.4				
III	0.6				
IV	0.8				
V	1.0				

<u>**Calculations**</u>

Molecular weight of the PVA

Mark – Hownik equation is given by

$$\eta_i = kM^a$$

$$\log \eta_i = \log k + a \log M$$

$$\log M = \left[\frac{\log \eta i - \log k}{a}\right]$$

$$M = A . \log \left[\frac{\log \eta i - \log k}{a}\right]$$

Where, M = Molecular weight of the polymer.

$$M = A \log \left[\frac{\log \ldots (-) \log (46.3) \times 10^{-6}}{0.64}\right]$$

<u>**RESULT**</u>

The molecular weight of the given polymer = ………….

EX. NO: 7
DATE:

ESTIMATION OF IRON CONTENT OF THE WATER BY SPECTROPHOTOMETRY

AIM

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

PRINCIPLE

Spectrophotometer is an instrument used to measure the intensity of the light absorbed by a substance. The relationship between absorbance (A) and the concentration of the solution (C) is given by Beer – Lambert's law.

$$\log I_0/I = A = \varepsilon Cx$$

Where

I_o = Intensity of incident light

I = Intensity of transmitted light

ε = Molar absorption co-efficient

x = Thickness of the cell

C = Concentration of the solution

From the equation it is seen that, the absorbance (A) is directly proportional to the molar concentration and thickness of the cell.

In the determination of the iron, ferrous iron solution is acidified with HNO_3 to convert Fe^{2+} into Fe^{3+}. Fe^{3+} ions does not give any colour in solution. But it gives red colour when it reacts with potassium thiocyanate (KSCN) (or) Ammonium thiocyanate (NH_4SCN) solution.

$$Fe^{3+} + 6\ KSCN \longrightarrow [Fe(SCN)_6]^{3-} + 6K^+$$
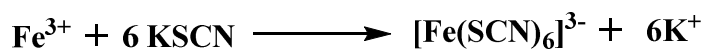
Blood red colour complex

This complex has maximum absorption in the region λ = 480 nm. A calibration curve is drawn by measuring the absorbance of known solution. Then the absorbance of unknown solution is measured, using which the concentration can be determined from the calibration curve.

MATERIALS REQUIRED

1. Spectrophotometer, 2. Ferrous ammonium sulphate, 3. HNO_3, 4. 25 ml standard flask 10 Nos, 5. Graduated pipette, 6. Potassium thiocyanate (or) Ammonium thiocyanate.

PROCEDURE

1. Preparation of stock Fe^{3+} iron solution

A stock solution of Fe^{3+} is prepared by dissolving 0.0838 gms of ferrous ammonium sulphate with 1 ml of con. HNO_3 and made upto 1 litre in a standard flask using distilled water.

From the stock solution, various concentrations are prepared (1, 2, 3, 4, 5, & 6 ppm) as shown in Table I.

(or)

Instead of various ppm Fe^{3+} solutions, various normal solutions are prepared.

A stock solution of 0.1 N Fe^{3+} is prepared by dissolving 39.0 gms of FAS with 1 ml of con. HNO_3 in 1 liter of distilled water.

From the stock solution, various concentrations are prepared (0.01, 0.02, 0.03, 0.04, 0.05 & 0.06 N) as shown in Table I.

2. Estimation of iron content

The spectrophotometer is switched on and warmed up to 10 minutes. The monochromater is adjusted for λ = 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 is 100.

Now the absorbance of all the standard solutions are similarly measured. Also the absorbance of unknown solution is measured. (Table II).

The calibration graph is drawn between the concentration and absorbance from which the concentration of unknown solution is measured.

Layout of spectrophotometer
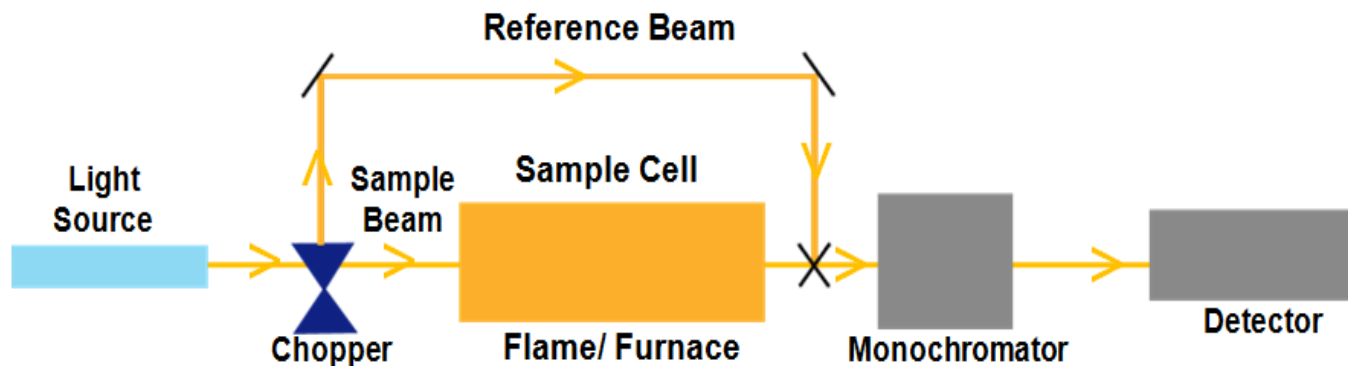
TABLE –I

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

S. No.	Volume Iron Solution ml	Volume of dil. HNO_3 ml	Volume of NH_4SCN ml	Volume of distilled H_2O (to make equal. Volume) ml	Concentration of Iron	
					ppm	N
1	10	5	10	75	1	0.01
2	20	5	10	65	2	0.02
3	30	5	10	55	3	0.03
4	40	5	10	45	4	0.04
5	50	5	10	35	5	0.05
6	60	5	10	25	6	0.06

Graph: Calibration curve (absorbance *Vs* concentration)
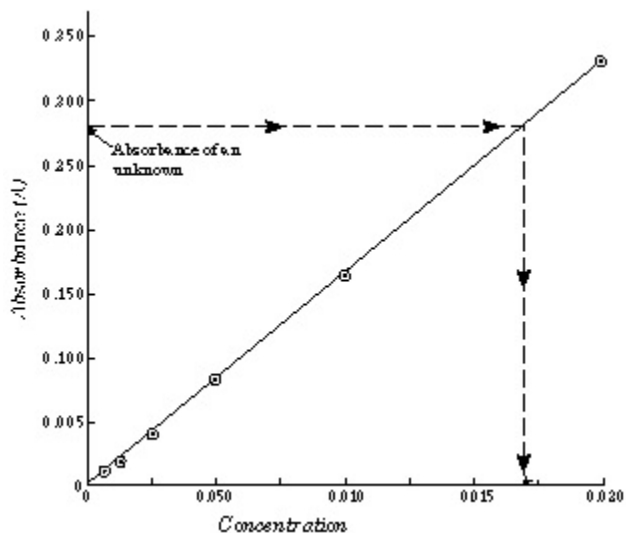
TABLE – II

Measurement of absorbance

Blank (distilled water): zero absorbance; λ_{max} = 480 nm

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

Calculation of amount of iron content

Amount of iron present in the given solution   **= N x Eq. wt of iron x 1000 ppm**

= ……… x 55.85 x 1000 ppm

= ……… ppm (or) mgs/lit

RESULT

Amount of iron present in the given water sample = ………… **ppm.**