



# Varuwan Vadivelan Institute of Technology

Dharmapuri – 636 703

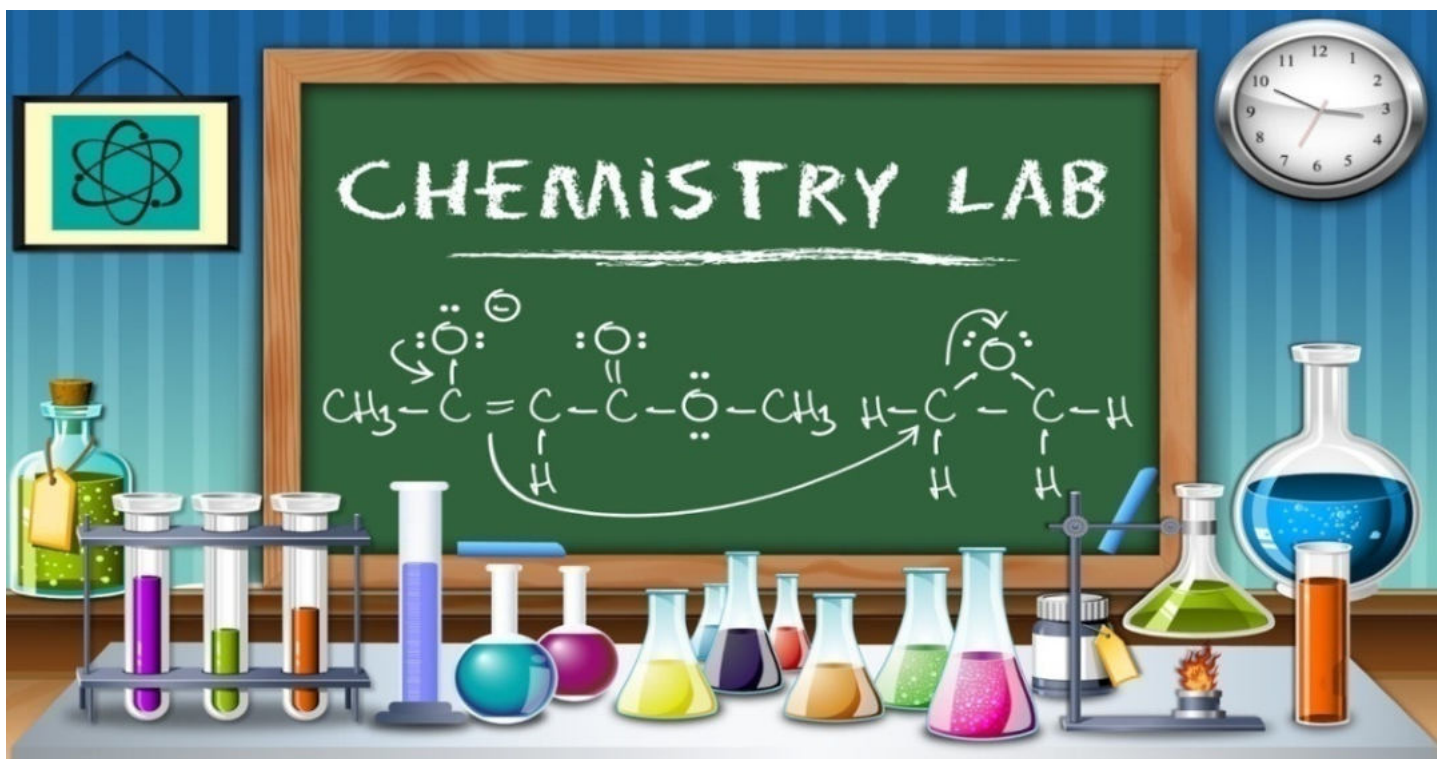
## LAB MANUAL

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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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:

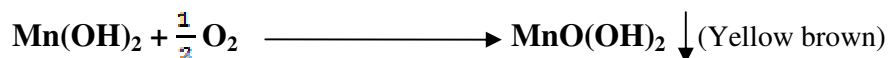
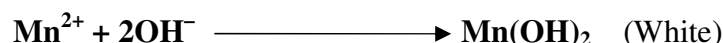
## DETERMINATION OF DISSOLVED OXYGEN IN WATER [Winkler's Method]

AIM

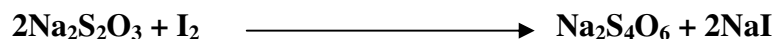
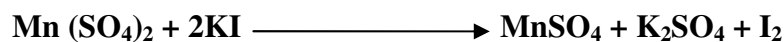
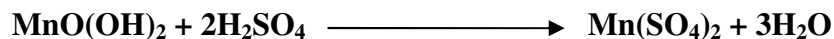
To determine the amount of dissolved oxygen in the given water sample by Winkler's method, a standard solution of  $K_2Cr_2O_7$  of strength .....N is given.

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.



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

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 shaken 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	$V_1 = 20 \text{ ml}$
Strength of Potassium dichromate	$N_1 = \dots\dots\dots N$
Volume of Sodium thiosulphate	$V_2 = \dots\dots\dots \text{ml}$
Strength of Sodium thiosulphate	$N_2 = ?$

According to the law of volumetric analysis

$$V_1 N_1 = V_2 N_2$$

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

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

Strength of Sodium thiosulphate =  $\dots\dots\dots N$

**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**  $V_1 = \dots\dots\dots$ mlStrength of **Sodium thiosuphate**  $N_1 = \dots\dots\dots$ NVolume of **Water sample**  $V_2 = 20$  mlStrength of **Water sample**  $N_2 = ?$ 

According to the law of volumetric analysis

$$V_1 N_1 = V_2 N_2$$

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

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

Strength of Water sample =  $\dots\dots\dots$ N

$$\begin{aligned}\text{Amount of dissolved oxygen in 1 litre of tap water} &= \text{Normality} \times \text{Eq.wt. of O}_2 \times 1000 \text{ mgs} \\ &= \dots\dots\dots N \times 8 \times 1000 \\ &= \dots\dots\dots \text{mgs/lit}\end{aligned}$$

**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].

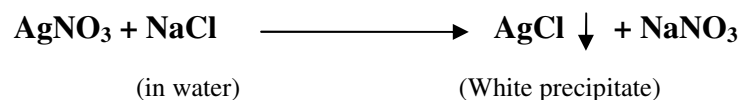
### AIM

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.

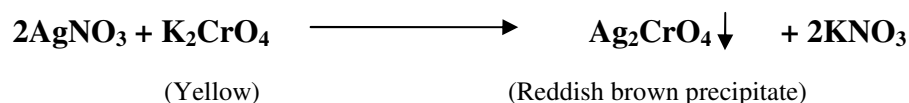
### PRINCIPLE

Generally water contains chloride ions ( $\text{Cl}^-$ ) in the form of NaCl, KCl,  $\text{CaCl}_2$  and  $\text{MgCl}_2$ . 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  $\text{Cl}^-$  ion solution is directly titrated against  $\text{AgNO}_3$  using potassium chromate ( $\text{K}_2\text{CrO}_4$ ) as an indicator.



At the end point, when all the  $\text{Cl}^-$  ions are removed. The yellow colour of chromate changes into reddish brown due to the following reaction.



### MATERIALS REQUIRED

1. Std. NaCl solution, 2.  $\text{AgNO}_3$  solution, 3. 2%  $\text{K}_2\text{CrO}_4$  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  $\text{AgNO}_3$  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  $\text{NaCl}$  solution. 20 ml of this solution is pipetted out into a clean conical flask. 1ml of 2%  $\text{K}_2\text{CrO}_4$  indicator is added and titrated against  $\text{AgNO}_3$  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%  $\text{K}_2\text{CrO}_4$  indicator is added. It is then titrated against standardized  $\text{AgNO}_3$  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<sub>3</sub>

Sl. No	Volume of NaCl ml	Burette Readings		Concordant volume of AgNO <sub>3</sub> ml	Indicator
		Initial ml	Final ml		
1.	20	0			K <sub>2</sub> CrO <sub>4</sub>
2.	20	0			

**CALCULATION:**Volume of Sodium Chloride  $V_1 = 20$  mlStrength of Sodium Chloride  $N_1 = \dots\dots\dots N$ Volume of Silver Nitrate  $V_2 = \dots\dots\dots ml$ Strength of Silver Nitrate  $N_2 = ?$ 

According to the law of volumetric analysis

$$V_1 N_1 = V_2 N_2$$

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

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

 $\therefore$  Strength of Silver Nitrate =  $\dots\dots\dots N$

**Step II: DETERMINATION OF CHLORIDE ION****TITRATION – II**  
Water sample Vs. Std. AgNO<sub>3</sub>

Sl. No	Volume of water sample ml	Burette Readings		Concordant volume of AgNO <sub>3</sub> ml	Indicator
		Initial ml	Final ml		
1.	20	0			K <sub>2</sub> CrO <sub>4</sub>
2.	20	0			

**Calculation of Normality of the water sample (Chloride ion)**

Volume of water sample	V <sub>1</sub> = 20 ml
Strength of water sample	N <sub>1</sub> = ?
Volume of Silver Nitrate	V <sub>2</sub> = .....ml
Strength of Silver Nitrate	N <sub>2</sub> = .....N

According to the law of volumetric analysis

$$V_1 N_1 = V_2 N_2$$

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

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

Strength of water sample = .....N

**Calculation of amount of the chloride**

$$\left. \begin{array}{l} \text{Amount of the chloride ion present in} \\ \text{1 litre of the water sample} \end{array} \right\} = \left\{ \begin{array}{l} \text{Equivalent weight of chloride ion X} \\ \text{Strength of the chloride ion} \end{array} \right.$$

$$= 35.46 \times \dots\dots\dots N$$

$$= \dots\dots\dots \text{gms}$$

- Amount of the chloride ion present in 100 ml of the given water sample

$$= \dots\dots\dots \text{gms} \times 100/1000$$

$$= \dots\dots\dots \text{gms}$$

**RESULT**

1. Amount of chloride ion present in the whole of the given water sample =  $\dots\dots\dots$ gms/mgs/ppm

2. Amount of chloride ion present in the 100 ml of the given water sample =  $\dots\dots\dots$ gms/mgs/ppm

EX. NO: 3

DATE:

**pH METRY - DETERMINATION OF STRNGTH OF HCl BY NaOH****AIM**

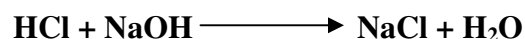
To determine the strength of given HCl by pH metry, a standard solution of NaOH of .....N is provided.

**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.



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.

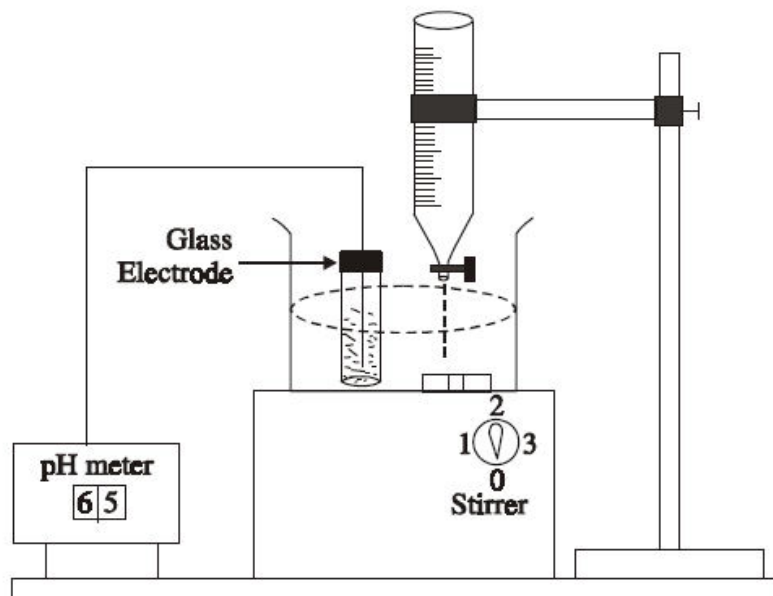
**PROCEDURE****TITRATION – I**

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  $\Delta\text{pH}/\Delta V$  against volume of NaOH added (graph II). From the end point, the strength of HCl solution and hence the amount of HCl is calculated.



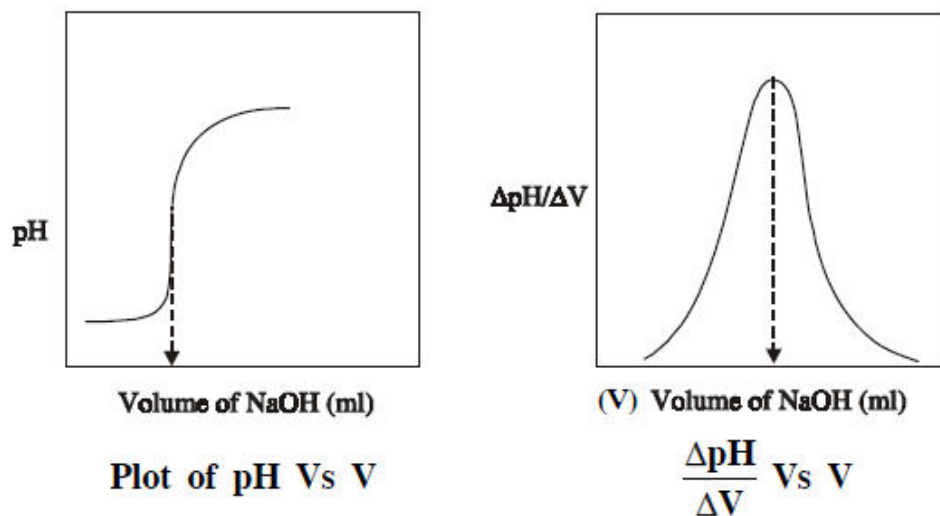
**Determination of pH**

**TABLE – I (Titration – I)**  
**Titration of HCl (20 ml) Vs. NaOH**

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

**TABLE - II (Titration - II)**  
**Titration of HCl (20 ml) Vs. NaOH**

S. No	Volume of NaOH ml	pH	$\Delta$ pH	$\Delta$ V MI	$\Delta$ pH / $\Delta$ V
1.					
2.					
3.					
4.					
5.					
6.					
7.					
8.					
9.					
10.					
11.					
12.					
13.					
14.					
15.					
16.					
17.					
18.					
19.					
20.					

**Model Graph****Calculation****Step I: Calculation of Strength of HCl**

Volume of HCl	$V_1 = 20 \text{ ml}$
Strength of HCl	$N_1 = ?$
Volume of NaOH	$V_2 = \dots\dots\dots \text{ml}$ (titre value)
Strength of NaOH	$N_2 = \dots\dots\dots \text{N}$

According to the law of volumetric analysis,

$$V_1 N_1 = V_2 N_2$$

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

$$= \frac{\dots\dots\dots \times \dots\dots\dots \text{N}}{20}$$

Strength of HCl =  $\dots\dots\dots \text{N}$

**Step – II: Calculation of amount of HCl**

$$\begin{aligned} \text{The amount of HCl present in 1000 ml of the given solution} &= \dots\dots\dots \text{N} \times \text{Eq. wt. of HCl (36.45)} \\ &= \dots\dots\dots \times 36.45 \\ &= \dots\dots\dots \text{gms} \end{aligned}$$

**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,



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 as 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.

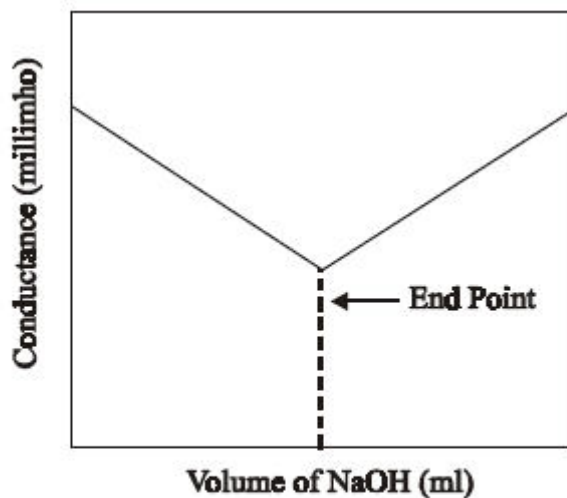
Now the graph is plotted by taking volume of NaOH in the X-axis and conductance in the Y-axis. The end point is the intersection of the two curves. From the volume of NaOH, the strength and hence the amount of NaOH present in 250 ml of the solution is calculated.

Step I

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

S. No	Volume of NaOH added		Conductance		Remarks
	ml		mho		
1.	0	21			Decreases           End point ●
2.	1	22			
3.	2	23			
4.	3	24			
5.	4	25			
6.	5	26			
7.	6	27			
8.	7	28			
9.	8	29			
10.	9	30			
11.	10	31			Increases
12.	11	32			
13.	12	33			
14.	13	34			
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	$V_1 = 20 \text{ ml}$
Strength of HCl	$N_1 = \dots\dots\dots N$
Volume of NaOH	$V_2 = \dots\dots\dots \text{ml}$ (titre value)
Strength of NaOH	$N_2 = ?$

According to the law of volumetric analysis,

$$V_1 N_1 = V_2 N_2$$

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

$$N_2 = \frac{20 \times \dots\dots\dots}{\dots\dots\dots}$$

$$\text{Strength of NaOH} = \dots\dots\dots N$$

**Calculation of amount of NaOH**

The amount of NaOH present in 1000 ml of the given solution =  $40 \times \dots\dots\dots N$   
=  $\dots\dots\dots \text{gms}$

The amount of NaOH present in 250 ml of the given solution =  $40 \times \dots\dots\dots N \times 250/1000$   
=  $\dots\dots\dots \text{gms}$

**RESULT**

1. The strength of given NaOH solution =  $\dots\dots\dots N$
2. The amount of NaOH present in 250 ml of the given solution =  $\dots\dots\dots \text{gms}$ .

EX. NO: 5

DATE:

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

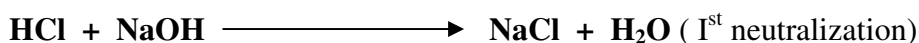
### AIM

To determine the strength and amount of a strong acid and a weak acid (HCl and CH<sub>3</sub>COOH) 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<sup>+</sup> ions are replaced by slow moving sodium ions, decrease in conductance take place until the end point is reached.



After the complete neutralization of all HCl, the neutralization of CH<sub>3</sub>COOH starts,



Since CH<sub>3</sub>COONa is stronger electrolyte than CH<sub>3</sub>COOH, conductivity slowly increases until all CH<sub>3</sub>COOH 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<sup>-</sup> ions.

### MATERIALS REQUIRED

1. Conductivity bridge, 2. Conductivity cell, 3. 100 ml beaker, 4. Standard NaOH, 5. Given HCl and CH<sub>3</sub>COOH mixture, 6. Burette, Pipette, glass rod etc., 7. Distilled water.

## PROCEDURE

### TITRATION

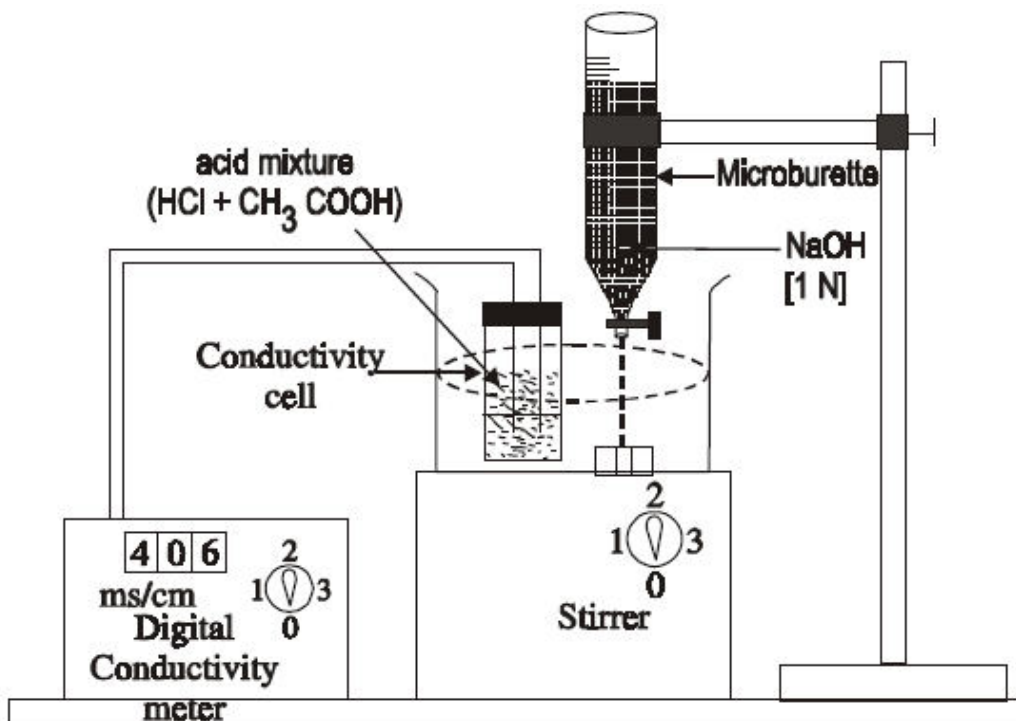
The given mixture of acids ( $\text{HCl} + \text{CH}_3\text{COOH}$ ) is transferred into 100 ml standard flask and made upto zero mark using distilled water. 20 ml of the made up solution is pipetted out into a clean 100 ml beaker and the conductivity cell is placed in it and immersed in the solution. 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  $\text{CH}_3\text{COOH}$  present in 1 liter of the mixture of solution are calculated.



Model graph

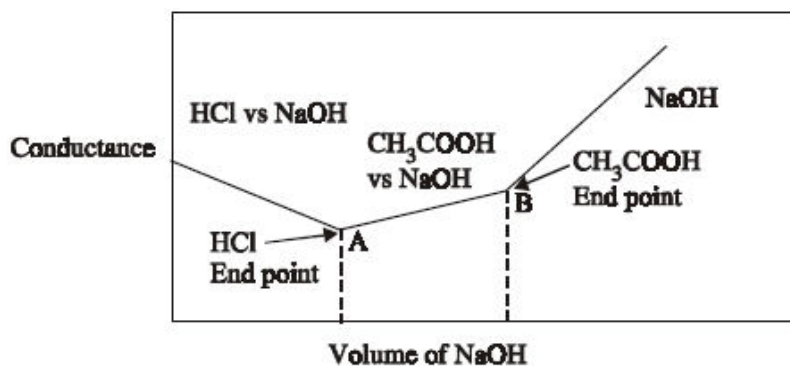


TABLE – I

Titration of mixture (HCl + CH<sub>3</sub>COOH) Vs. NaOH

S. No	Volume of NaOH added		Conductance	
	ml		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)	$V_1 = 20 \text{ ml}$
Strength of the mixture (HCl)	$N_1 = ?$
Volume of the NaOH	$V_2 = \dots\dots\dots (\text{A}) \text{ ml}$ (I <sup>st</sup> titre value)
Strength of the NaOH	$N_2 = \dots\dots\dots \text{N}$

According to the law of volumetric analysis,

$$V_1 N_1 = V_2 N_2$$

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

$$= \frac{\dots\dots\dots \text{ml} \times \dots\dots\dots \text{N}}{20}$$

$$\text{Strength of HCl} = \dots\dots\dots \text{N}$$

**Calculation of amount of HCl**

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

$$= \text{Strength of HCl} \times \text{Eq. wt. of HCl}$$

$$= \dots\dots\dots \text{N} \times 36.45$$

$$= \dots\dots\dots \text{gms}$$

**Step I****Calculation of Strength of CH<sub>3</sub>COOH**

Volume of the mixture (CH <sub>3</sub> COOH)	$V_1 = 20 \text{ ml}$
Strength of the mixture (CH <sub>3</sub> COOH)	$N_1 = ?$
Volume of the NaOH	$V_2 = \dots\dots\dots (\text{B-A}) \text{ ml}$ (II <sup>nd</sup> titre value)
Strength of the NaOH	$N_2 = \dots\dots\dots \text{N}$

According to the law of volumetric analysis,

$$V_1 N_1 = V_2 N_2$$

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

$$= \frac{\dots\dots\dots \text{ml} \times \dots\dots\dots \text{N}}{20}$$

Strength of  $\text{CH}_3\text{COOH}$  =  $\dots\dots\dots$  N

### Calculation of amount of $\text{CH}_3\text{COOH}$

$$\therefore \text{The amount of } \text{CH}_3\text{COOH} \text{ present in } \left. \begin{array}{l} 1000 \text{ ml of the given solution} \end{array} \right\} = \text{Strength of } \text{CH}_3\text{COOH} \times \text{Eq.wt. of } \text{CH}_3\text{COOH}$$

$$= \dots\dots\dots \text{N} \times 60$$

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

### RESULT

1. The strength of HCl present in 1 litre of the given solution =  $\dots\dots\dots$  N
2. The amount of HCl present in 1 litre of the given solution =  $\dots\dots\dots$  gms
3. The strength of  $\text{CH}_3\text{COOH}$  present in 1 litre of the given solution =  $\dots\dots\dots$  N
4. The amount of  $\text{CH}_3\text{COOH}$  present in 1 litre of the given solution =  $\dots\dots\dots$  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 ( $\eta_i$ ) 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.

Molecular 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 ( $\eta_r$ )  $\eta_r = \frac{\eta}{\eta_0} = \frac{t}{t_0}$

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

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

$$4. \text{ Intrinsic viscosity } (\eta_i) \quad \eta_i = \lim_{c \rightarrow 0} \frac{\eta_{sp}}{c}$$

Where,

$\eta_i$  = Absolute viscosity of a PVA solution.

$\eta_o$  = Absolute viscosity of a pure solvent

$t$  = Flow time for the PVA solution

$t_o$  = Flow time for the solvent (H<sub>2</sub>O).

## **PROCEDURE**

### **Step I: Preparation of PVA solutions 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**

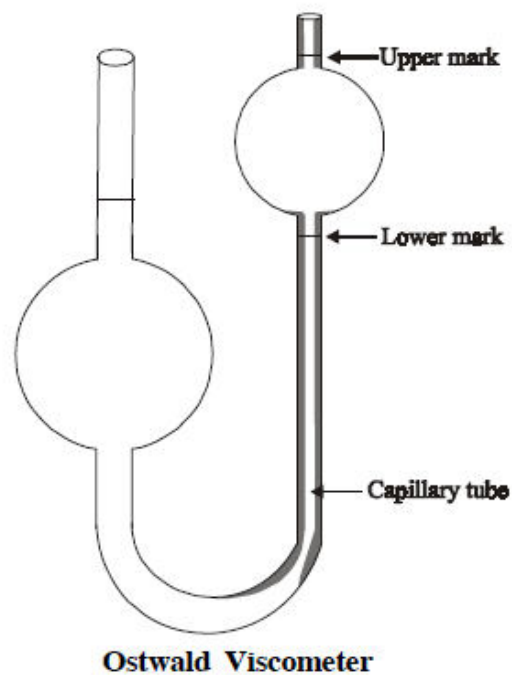
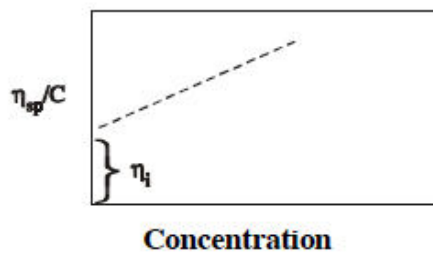
20 ml of the water is taken into the viscometer and is sucked through the capillary tube upto the uppermark, without any air bubbles. Now the flow time is noted for the water to flow from the uppermark (A<sub>1</sub>) to lowermark (A<sub>2</sub>).

### **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 (II<sup>nd</sup>, III<sup>rd</sup> etc.,) are determined.

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

From the flow times, reduced viscosity ( $\eta_{sp}/C$ ) can be calculated. Graph is plotted between  $\eta_{sp}/C$  vs concentration, straight line is obtained with an intercept is called intrinsic viscosity ( $\eta_i$ ).

**Model Graph****TABLE - I****Preparation of various PVA solutions**

S. No	Volume of 1% PVA solutions (Stock Solutions)	Volume of water	Concentration
	ml	ml	%
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

**TABLE – II**

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				

**Calculations****Molecular weight of the PVA**

Mark – Hownik equation is given by

$$\eta_i = kM^a$$

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

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

$$M = A \cdot \log \left[ \frac{\log \eta_i - \log k}{a} \right]$$

Where, M = Molecular weight of the polymer.

$$M = A \log \left[ \frac{\log \eta_i - \log k}{0.64} \right]$$

**RESULT**

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.

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

Where

$I_0$  = Intensity of incident light

$I$  = Intensity of transmitted light

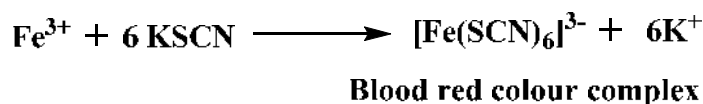
$\epsilon$  = Molar absorption co-efficient

$x$  = 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  $\text{HNO}_3$  to convert  $\text{Fe}^{2+}$  into  $\text{Fe}^{3+}$ .  $\text{Fe}^{3+}$  ions does not give any colour in solution. But it gives red colour when it reacts with potassium thiocyanate (KSCN) (or) Ammonium thiocyanate ( $\text{NH}_4\text{SCN}$ ) solution.



This complex has maximum absorption in the region  $\lambda = 480$  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<sub>3</sub>, 4. 25 ml standard flask 10 Nos, 5. Graduated pipette, 6. Potassium thiocyanate (or) Ammonium thiocyanate.

### **PROCEDURE**

#### **1. Preparation of stock Fe<sup>3+</sup> iron solution**

A stock solution of Fe<sup>3+</sup> is prepared by dissolving 0.0838 gms of ferrous ammonium sulphate with 1 ml of con. HNO<sub>3</sub> 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<sup>3+</sup> solutions, various normal solutions are prepared.

A stock solution of 0.1 N Fe<sup>3+</sup> is prepared by dissolving 39.0 gms of FAS with 1 ml of con. HNO<sub>3</sub> 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**

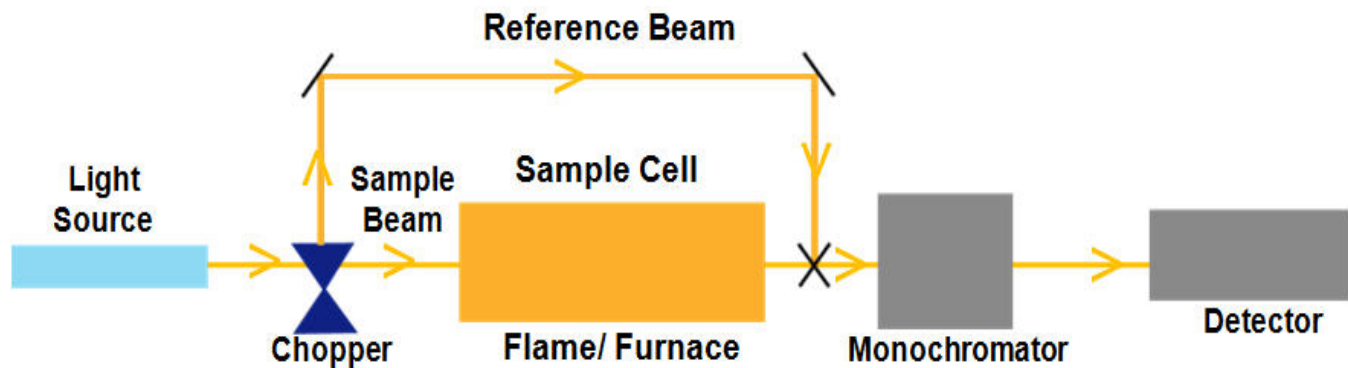
The spectrophotometer is switched on and warmed up to 10 minutes. The monochromater is adjusted for  $\lambda = 480$  nm.

The blank solution (distilled water) is kept in the cell and the absorbance is measured for which the absorbance is zero and transmittance 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**

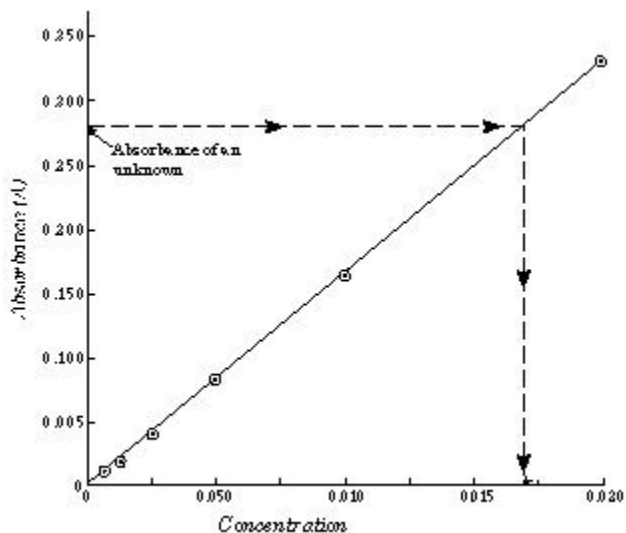


**TABLE -I**

**Preparation of various concentration of Fe<sup>3+</sup> solution**

S. No.	Volume Iron Solution ml	Volume of dil. HNO <sub>3</sub> ml	Volume of NH <sub>4</sub> SCN ml	Volume of distilled H <sub>2</sub> O (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;  $\lambda_{\max} = 480 \text{ nm}$

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**

$$\begin{aligned} \text{Amount of iron present in the given solution} &= N \times \text{Eq. wt of iron} \times 1000 \text{ ppm} \\ &= \dots\dots\dots \times 55.85 \times 1000 \text{ ppm} \\ &= \dots\dots\dots \text{ ppm (or) mgs/lit} \end{aligned}$$

**RESULT**

Amount of iron present in the given water sample =  $\dots\dots\dots$  ppm.