

www.studentsfocus.com

Department of Chemistry

(2015-2016)

GE 6163 – CHEMISTRY LAB MANUAL

Step I : Standardization of sodium thiosulphate

Titration I (Standard dichromate vs Sodium thiosulphate)

Sl.No	Volume of Potassium dichromate (ml)	Burette reading (ml)		Volume of Sodium thiosulphate (ml)	Indicator
		Initial	Final		
1	20	0		V ₁	Starch
2	20	0			
3	20	0			

Volume of Potassium dichromate (V₁) = 20 ml

Strength of Potassium dichromate (N₁) = 0.0125 N

Volume of Sodium thiosulphate (V₂) = --- ml

Strength of Sodium thiosulphate (N₂) = --- N

According to volumetric formula

$$V_1 N_1 = V_2 N_2$$

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

Strength of Sodium thiosulphate (N₂) = --- N

1. ESTIMATION OF DISSOLVED OXYGEN OF BOILER FEED WATER

Expt. No.	
-----------	--

Date			
------	--	--	--

AIM

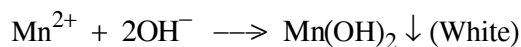
To determine the dissolved oxygen in the boiler feed water.

CHEMICALS REQUIRED

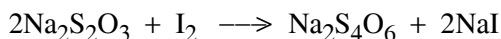
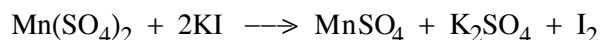
$\text{Na}_2\text{S}_2\text{O}_3$ (N/40), MnSO_4 solution, KI, starch, conc. H_2SO_4

PRINCIPLE

Oxygen dissolves in water to the extent of 7 - 9 mgs/lit at a temperature range of 25 - 35°C. The estimation of dissolved oxygen in water is useful in studying water pollution. Water sample is collected carefully avoiding aeration/deaeration in ground stoppered flask. Initially manganous sulphate and alkali-iodide reagents are added and the reaction occurs as follows



Potassium iodide and the precipitate react with concentrated sulphuric acid liberating iodine and the liberated iodine is titrated against $\text{Na}_2\text{S}_2\text{O}_3$



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. 20 ml of 0.0125N potassium dichromate solution is pipetted out into a clean conical flask. To this, 5 ml of sulphuric acid and 50 ml of 5% potassium iodide 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 values.

Step II : Estimation of dissolved oxygen

Titration II (Water sample vs Sodium thiosulphate)

Sl.No	Volume of Potassium dichromate (ml)	Burette reading (ml)		Volume of Sodium thiosulphate (ml) V_2	Indicator
		Initial	Final		
1	20	0			Starch
2	20	0			
3	20	0			

Volume of Sodium thiosulphate (V_1) =

Strength of Sodium thiosulphate (N_1) =

Volume of water sample (V_2) = 100 ml

Strength of water sample (N_2) = --- N

According to volumetric formula

$$V_1 N_1 = V_2 N_2$$

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

Amount of dissolved oxygen in one litre of tap water =

Normality \times Eq.wt.of $O_2 \times 1000$ mg.

$$= \text{---} N \times 8 \times 1000$$

$$= \text{---} \text{ mg/lit}$$

Titration II

Estimation of dissolved oxygen

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

The stopper is replaced and the flask is inverted several times for complete dissolution of the precipitate. 100 ml of the brown coloured solution is pipetted out and titrated against standardized sodium thiosulphate solution. Starch indicator is added when the solution becomes light yellow. The titration is continued until the blue colour disappears. From the titre value, the strength of dissolved oxygen and hence the amount of dissolved oxygen in the water sample is calculated.

RESULT

Amount of dissolved oxygen in water sample = ----- mg/lit

Titration 1 : Standardisation of silver nitrate

Std. NaCl vs AgNO₃

Sl. No	Volume of std. NaCl (ml)	Burette reading (ml)		Volume of AgNO ₃ (V ₁ ml)	Concordant value (ml)	Indicator
		Initial	Final			
1.	20	0				K ₂ CrO ₄
2.	20	0				

Calculation of the Strength of silver nitrate

Volume of std. NaCl (V₂) = 20 ml

Strength of NaCl (N₂) = 0.01 N

Volume of AgNO₃ (V₁) = ____ ml

Strength of AgNO₃ (N₁) = ?

According to volumetric formula

$$V_1 N_1 = V_2 N_2 \text{ i.e. } N_1 = V_2 \times \frac{N_2}{V_1}$$

$$N_1 = 20 \text{ ml} \times 0.01 \text{ N} / V_1$$

Strength of AgNO₃ (N₁) = _____ N

2. ESTIMATION OF CHLORIDE CONTENT IN WATER BY ARGENTOMETRIC METHOD (MOHR'S METHOD)

Expt. No.	
-----------	--

Date			
------	--	--	--

AIM

To estimate the amount of chloride ion present in the water sample by Argentometric method (Mohr's method). You are provided with standard NaCl solution of strength 0.01 N and a link solution of AgNO₃ (approximately 0.01N).

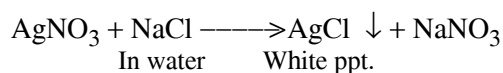
CHEMICALS REQUIRED

Standard NaCl solution, AgNO₃ solution, Potassium chromate indicator

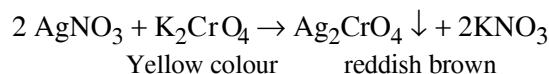
PRINCIPLE

Natural water contains chloride ions in the form of NaCl, KCl, CaCl₂, MgCl₂. The concentration of chloride ions more than 250 ppm is not desirable for drinking purposes.

This determination is based on precipitation titration. When AgNO₃ solution is added to the water sample, in presence of K₂CrO₄, the chlorides present in it are precipitated first as AgCl.



When all the Cl⁻ ions is removed, AgNO₃ added from the burette will react with K₂CrO₄ to give a reddish brown colour due to silver chromate (Ag₂CrO₄). This is the end point.



PROCEDURE

Titration 1: Standardisation of AgNO₃

The burette is washed well with distilled water and rinsed with small amount of AgNO₃ solution. The pipette is washed with distilled water and rinsed with small amount of standard NaCl solution. 20ml of this solution is pipetted out into a clean conical flask. 1ml of 2% K₂CrO₄ indicator solution is added and titrated against AgNO₃ 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.

Titration 2 : Estimation of Chloride

Water sample vs Standard AgNO₃

Sl. No	Volume of water sample (ml)	Burette reading (ml)		Volume of AgNO ₃ (ml)	Concordant value (ml)	Indicator
		Initial	Final			
1.	20	0				K ₂ CrO ₄
2.	20	0				

Calculation of the normality of water sample (Chloride ion)

Volume of water sample (V₁) = 20 ml

Strength of water sample (N₁) = ?

Volume of AgNO₃ (V₂) = ____ ml

Strength of AgNO₃ (N₂) = ____ N

According to volumetric formula

$$V_1 N_1 = V_2 N_2$$

$$N_1 = V_2 \times N_2 / 20$$

Strength of water sample (N₁) = ____ N

Calculation of amount of chloride

Amount of chloride present in 1 litre of the given water sample = Eq.wt of chloride ion × Normality of chloride ion

Amount of chloride ion present in 100 ml of the given water sample = $35.46 \times \text{Normality of chloride ion} \times 100/1000\text{gm}$

Titration 2: Estimation of Chloride ion

The given water sample is made up into 100 ml in a standard flask using distilled water. 20ml of this solution is pipetted out into a clean conical flask and 1ml of 2% K_2CrO_4 indicator solution is added. It is then titrated against standard $AgNO_3$ 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.

RESULT

The amount of chloride ion present in 100 ml of the given water sample = _____ gms.

3. pH METRY – DETERMINATION OF STRENGTH OF HCl

Expt. No.	
-----------	--

Date			
------	--	--	--

AIM :

To determine the strength of given hydrochloric acid by pH metry. You are provided with a standard solution of 0.1N sodium hydroxide.

MATERIALS REQUIRED

Hydrochloric acid, sodium hydroxide, pH meter, glass electrode

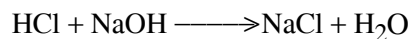
PRINCIPLE

pH of the solution is related to the H_3O^+ ion concentration of the solution by the expression

$$pH = -\log [H_3O^+]$$

The concentration of H^+ ions in the solution is determined by measuring the pH of the solution.

When NaOH is added slowly from the burette to the solution of HCl, H^+ ions are neutralized by OH^- ions. As a result, pH of the solution increases.



The increase in pH takes place until all the H^+ ions are completely neutralized. After the endpoint, further addition of NaOH increases the pH sharply as there is an excess of OH^- ions.

Model Graph

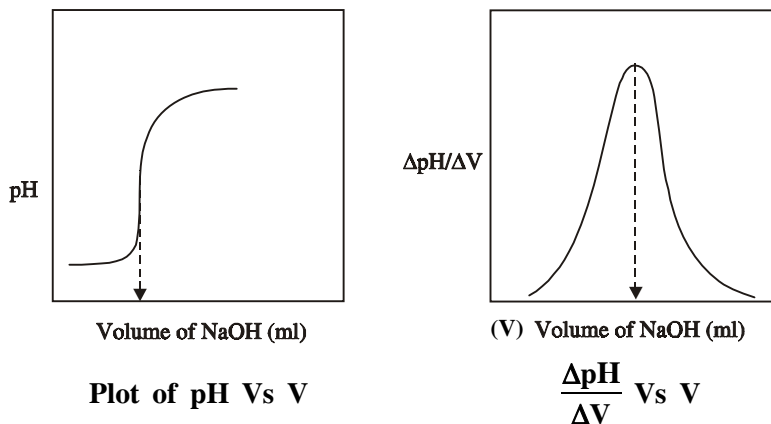


Table 2
Titration of HCl vs NaOH

S.No	Volume of NaOH (ml)	pH	Δ pH	Δ V (ml)	Δ pH/Δ V	V = (V ₁ + V ₂)/2

Calculation

Volume of HCl V₁ = 20 ml

Strength of HCl N₁ = _____ ?

Volume of NaOH V₂ = ___ ml

Strength of NaOH N₂ = 0.1 N

Strength of HCl N₁ = V₂N₂/V₁ = V₂ × 0.1/20

Weight of HCl in 1 litre of the given solution = ___ N₁ × eq.wt of HCl g
= N₁ × 36.5 gm/l

Weight of HCl in 100 ml of the given solution = N₁ × 36.5/10 gm.

PROCEDURE:

The burette is filled with standard NaOH solution. 20ml of HCl solution is pipetted out into a clean beaker. It is diluted to 100ml with distilled water. The glass electrode is dipped into the solution and connected to the pH meter.

The NaOH solution is gradually added from the burette to the HCl solution in the beaker. pH is noted after each addition. The observed pH values are plotted against the volume of NaOH added. From the graph the end point is determined.

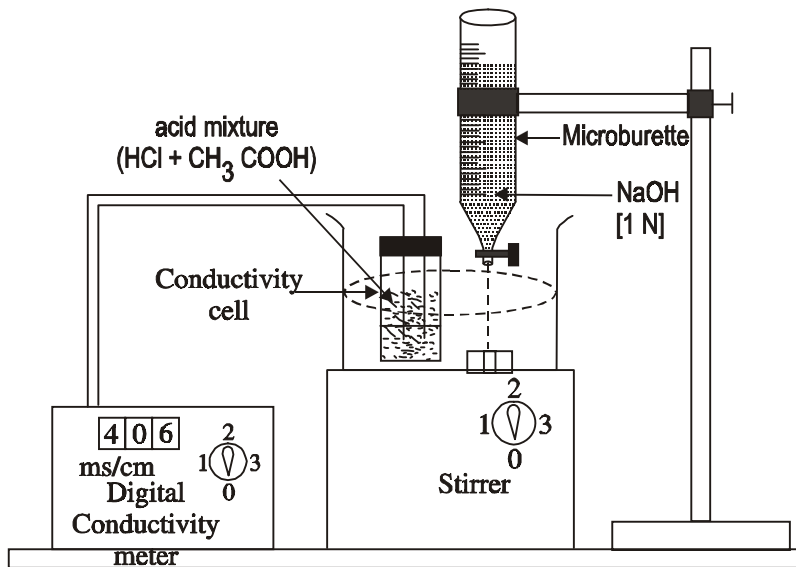
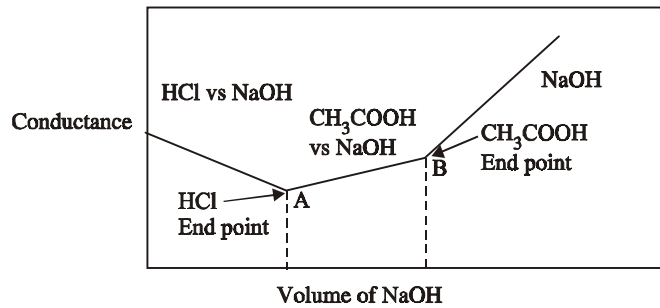
RESULT:

The amount of HCl present in the given solution is = g.

Table 1
Titration of mixture of acids vs NaOH

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

Model graph



4. CONDUCTOMETRIC TITRATION OF MIXTURE OF ACIDS

Expt. No.	
------------------	--

Date			
-------------	--	--	--

AIM:

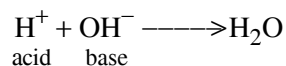
To estimate the amount of hydrochloric (HCl) and acetic acids (CH₃COOH) present in the given mixture. You are provided with a standard solution of NaOH of strength 0.5 N.

MATERIALS REQUIRED:

Conductivity meter, conductivity cell, microburette, pipette, Std. 0.5N NaOH.

PRINCIPLE:

Conductivity of a solution depends upon the number and nature of ions in solution. During the addition of base to acid solution, the H⁺ ions are removed as water. Hence the conductance of the solution decreases.



In a mixture of strong acid and weak acid, strong acid reacts with the base initially. The decrease in conductivity is sharp. After the neutralization of strong acid, weak acid reacts with the added base. Acetic acid being weakly ionized, the decrease in conductivity is slow. At the end point, when all the acids are neutralized, the addition of base increases the OH⁻ ion in solution. Hence there is a sharp increase in conductivity.

PROCEDURE:

The burette is filled with sodium hydroxide (NaOH) solution up to the zero mark. 10ml of HCl and 10ml of CH₃COOH are taken in a 250ml beaker. The conductivity cell is placed in it and then diluted to 100ml with distilled water. It is then connected with the conductivity meter.

Then 0.5 ml of alkali is added from the burette to the solution. The solution is stirred carefully and then conductance is measured after each addition of alkali.

Calculation of strength of HCl

Volume of mixture (HCl)	$V_1 = 20 \text{ ml}$
Strength of mixture (HCl)	$N_1 = \text{_____} ?$
Volume of NaOH	$V_2 = \text{___} \text{ (A) ml (1}^{\text{st}} \text{ titre value)}$
Strength of NaOH	$N_2 = 0.5 \text{ N}$
	$N_1 = V_2 N_2 / V_1 = V_2 \times 0.5 / 20$
Strength of HCl	$N_1 = \text{_____} \text{ N}$

The amount of HCl present in

$$\begin{aligned} 1 \text{ litre of the given solution} &= \text{Strength of HCl} \times \text{Eq.wt. of HCl} \\ &= \text{_____} \text{ N} \times 36.5 \\ &= \text{_____} \text{ g/l} \end{aligned}$$

Calculation of strength of CH_3COOH

Volume of mixture (CH_3COOH)	$V_1 = 20 \text{ ml}$
Strength of mixture (CH_3COOH)	$N_1 = \text{_____} ?$
Volume of NaOH	$(B - A) V_2 = \text{_____} \text{ ml}$
Strength of NaOH	$N_2 = 0.5 \text{ N}$
	$N_1 = V_2 N_2 / V_1 = V_2 \times 0.5 / 20$
Strength of CH_3COOH	$N_1 = \text{_____} \text{ N}$

The amount of CH_3COOH present in

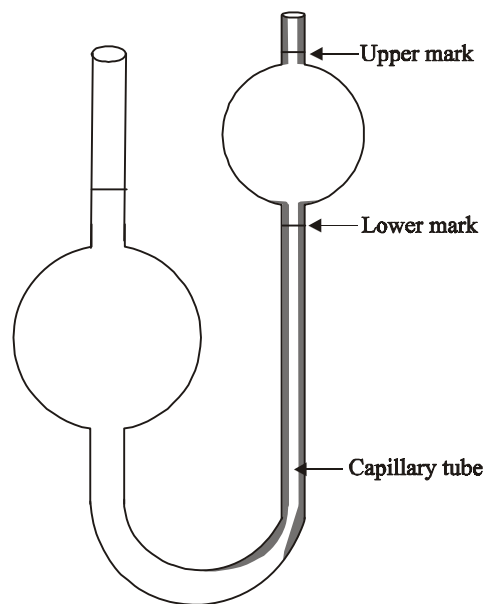
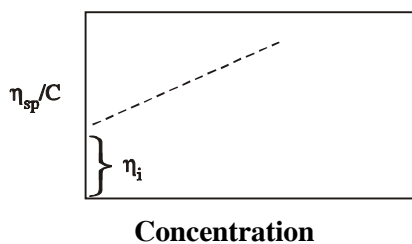
$$\begin{aligned} 1 \text{ litre of the given solution} &= \text{Strength of } \text{CH}_3\text{COOH} \times \text{Eq.wt. of } \text{CH}_3\text{COOH} \\ &= \text{_____} \text{ N} \times 60 \\ &= \text{_____} \text{ g/l} \end{aligned}$$

A graph is drawn between conductance and volume of NaOH. From the graph, the first end point A and the second end point B are noted. The amount of HCl and CH₃COOH present in 1 litre of the mixture is calculated from the end points A and B respectively.

RESULT

1. The amount of HCl present in 1 litre of the given solution = _____ g/l
2. The amount of CH₃COOH present in 1 litre of the given solution = _____ g/l

Model Graph



Ostwald Viscometer

Table 1
Preparation of polymer solutions of various dilutions
Polymer solutions are prepared in a 50ml standard flask

S. No	Volume of polymer solution (ml)	Volume of distilled water (ml)	Concentration (%)
1	The given 1 % solution	—	1%
2	$V_1N_1 = V_2N_2$ $V_2 = 50 \times 0.5/1 = 25$	25	0.5
3	$V_1N_1 = V_2N_2$ $V_2 = 50 \times 0.4/1 = 20$	30	0.4
4	$V_1N_1 = V_2N_2$ $V_2 = 50 \times 0.3/1 = 15$	35	0.3
5	$V_1N_1 = V_2N_2$ $V_2 = 50 \times 0.2/1 = 10$	40	0.2
6	$V_1N_1 = V_2N_2$ $V_2 = 50 \times 0.1/1 = 5$	45	0.1

6. DETERMINATION OF MOLECULAR WEIGHT OF A POLYMER

Expt. No.	
-----------	--

Date			
------	--	--	--

AIM

To determine the molecular weight of the polymer using Ostwald's viscometer. You are provided with 1% solution of the given polymer (Polyvinyl alcohol).

MATERIALS REQUIRED

Polymer solution, Viscometer, solvent, stop clock, 50 ml standard flasks

PRINCIPLE

Molecular weight of the polymer means average molecular weight of the polymer. This can be determined from intrinsic viscosity of a dilute polymer solution. The limiting value of reduced viscosity or inherent viscosity at infinite dilution of the polymer is known as intrinsic viscosity of polymer solution.

When a very small amount of polymer is added to a solvent of low viscosity, the viscosity of the resulting solution increases sharply. This increase in viscosity depends upon the molecular weight of the polymer, concentration, size and shape of the solute molecules.

The relative viscosity is the ratio of the viscosity of the solution to the viscosity of the solvent. This is given by

$$\eta_r = \frac{\eta_s}{\eta_o}$$

η_s is the coefficient of viscosity of the polymer solution

η_o is the coefficient of viscosity of pure solvent (water) at the same temperature.

For linear polymers, the intrinsic viscosity is related to the molecular weight of the polymer by Mark – Kuhn- Houwink equation:

$$[\eta] = kM^a$$

Where k and a are constants for a given polymer, solvent combination at a given temperature.

The intrinsic viscosity is related to the specific viscosity of a polymer solution by the relation

$$[\eta] = [\eta_{sp}/C]$$

η_{sp} - specific viscosity of the polymer. It is the relative viscosity of a polymer solution of known concentration minus 1.

Table 2
Viscosity data of polymer solution/ solvent

Flow time of pure solvent (t₀) = _____ sec

S. No.	Concentration of polymer	Flow time t (sec)			$\eta/\eta_0 = t/t_0 = \eta_r$	$\eta_{sp} = \eta_r - 1$	$\eta_{red} = \eta_{sp}/C$
		t ₁	t ₂	t (avg)			
1	1%						
2	0.5%						
3	0.4%						
4	0.3%						
5	0.2%						
6	0.1%						

Calculation

Mark – Kuhn- Houwink equation:

$$[\eta_i] = kM^a$$

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

$$\log M = \left(\frac{\log \eta_i - \log k}{a} \right)$$

$$M = \text{antilog} \left(\frac{\log \eta_i - \log k}{a} \right)$$

where M = Molecular weight of the polymer

For polyvinyl alcohol

$$k = 45.3 \times 10^{-5} \text{ gm/dl}$$

$$a = 0.64$$

$$\eta_{sp}/C = \text{Viscosity/ concentration in \%weight} = \text{viscosity} \times 100 / \text{concentration}$$

C – concentration of the solution

η_{sp} is related to η_r by

$$[\eta_{sp}] = \eta_r - 1$$

Where $\eta_r = \frac{t}{t_0}$

t and t_0 are flow times for solution and solvent respectively

$[\eta]$ can be known by drawing a graph between η_{sp}/C vs C. The intercept of η_{sp}/C at 0 concentration is known as intrinsic viscosity.

PROCEDURE

Step 1: Polymer solutions of different concentrations 1%, 0.5%, 0.4%, 0.3%, 0.2%, 0.1% are prepared from the given polymer stock solution as in table 1.

Step 2: The solvent is taken in the viscometer. It is sucked through the capillary tube upto the uppermark, without any air bubbles. The flow time of the solvent is noted as it flows from the upper mark to the lower mark. The flow time measurements is repeated twice and the average of the two readings are taken.

Step 3: The viscometer is first filled with the 1 % polymer solution. The flow time is measured as before. Similarly the flow times of other dilutions are also determined and tabulated.

For each polymer solution, the viscometer should be thoroughly washed and rinsed with the solvent.

From the flow times, reduced viscosity (η_{sp}/C) can be calculated. Graph is plotted between η_{sp}/C vs C. A straight line is obtained. The intercept is called intrinsic viscosity.

Degree of polymerisation:

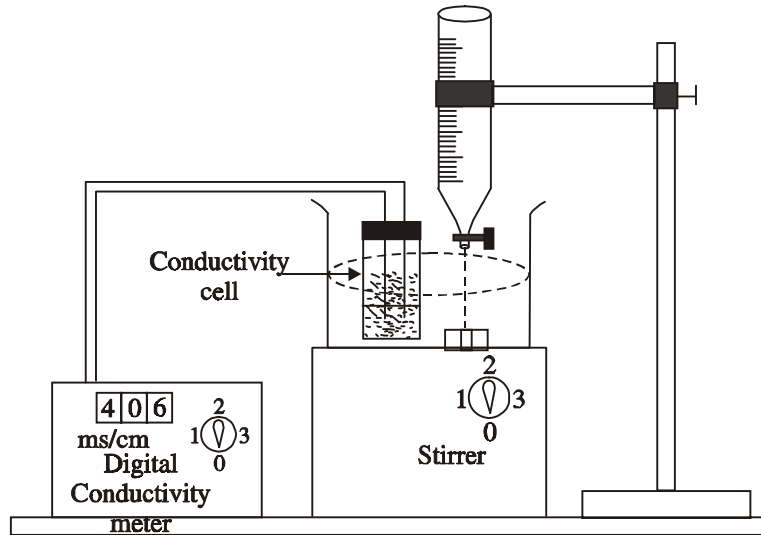
It is the number of repeating units in a polymer molecule. The repeating unit in PVA is $\left[\begin{array}{c} \text{CH}_2 - \text{CH} \\ | \\ \text{OH} \end{array} \right]$ corresponding to unit weight of 44.

The degree of polymerisation = $\frac{\text{Calculated M.wt of polymer}}{44}$ per molecule

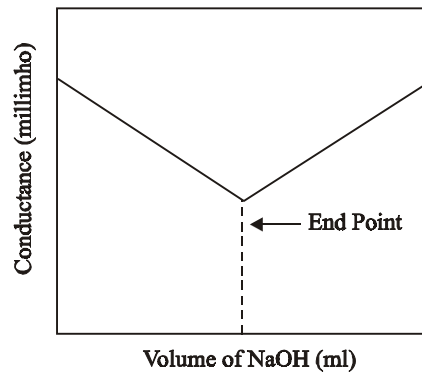
RESULT

The molecular weight of the given polymer = _____

The degree of polymerisation = _____



Determination of Conductance



HCl Vs NaOH

7. CONDUCTOMETRIC TITRATION OF STRONG ACID WITH STRONG BASE

Expt. No.	
-----------	--

Date			
------	--	--	--

Aim

To determine the amount of strong acid (HCl) present in the given sample by conductometric titration. You are provided with NaOH solution of strength 0.2N.

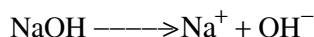
Principle

A solution of an electrolyte conducts electricity due to its dissociation into ions which depends on their number and mobility. Since specific conductance of a solution is proportional to the concentration of ions in it, conductance of the solution is measured during titration.

A reaction between strong acid and strong base, leading to the following neutralization.



This reaction is followed conductometrically in a conductivity bridge using a conductivity cell. When a strong base of 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 conductance of the solution decreases. This decrease in conductance will take place until the end point is reached. Further addition of NaOH increases the conductance sharply as there is an excess of fast moving OH^- ion.



A plot is made between volume of NaOH added and the conductance of solutions. The end point is intersection of the two lines.

Procedure

The microburette is filled with standard NaOH solution. 20 ml of the given HCl is pipetted out into a clean 100 ml beaker. The conductivity cell is placed in it and then diluted to 50 ml by adding conductivity water. The two terminals of the cell are connected to conductivity bridge. Initial conductance is read in the instrument for the acid alone without the addition of NaOH.

Now 0.05 ml of NaOH solution from the burette is added to the solution taken in the beaker, stirred and then conductivity is measured. The process is continued up to the end point. After the end point, further NaOH is gradually added and few more readings are noted.

Table

Sl.No.	Volume of NaOH added (ml)	Conductance (millimho)
1.		
2.		
3.		
4.		
5.		
6.		
7.		
8.		
9.		
10.		

Calculation:

Step - 1: Calculation of normality of HCl:

Volume of NaOH $V_1 = \text{_____}$ ml (titre value)

Strength of NaOH $N_1 = \text{_____}$ N

Volume of HCl $V_2 = 10$ ml

Strength of HCl $N_2 = ?$

By volumetric principle $V_1 N_1 = V_2 N_2$

$$= 20 \times N_2$$

$$N_2 = \frac{V_1 \times N_1}{10} = \text{_____} \text{ N}$$

\therefore Strength of HCl $N_2 = \text{_____}$ N

Step - 2: Calculation of amount of HCl

The amount of HCl present in the whole of the given solution } $= N_2 \times \text{Eq. wt. of HCl}$

$$= N_2 \times 36.45$$

$$= \text{_____} \text{ g/lit}$$

A graph is plotted between the volume of NaOH and conductance and the end point is noted. It is the intersection of the two lines as in the figure. The amount of HCl present in the given solution is calculated.

RESULT

Amount of the HCl present in the given acid = _____ g/lit