Ex. No. 1 DETERMINATION OF Na_2CO_3 AND NaOH IN A MIXTURE BY TITRATION

AIM:

To determine the amount of Na_2CO_3 and NaOH in a mixture using hydrochloric acid.

Principle:

When a known volume of the mixture is titrated with HCl in presence of phenolphthalein, the acid reacts with all the sodium hydroxide and with only half of the carbonate.

When the titration is continued with methyl orange indicator, the remaining half of CO_3^{2-} ions will be neutralized with HCl at the end point.

A	=	all hydroxide ions + half of carbonate ions
B	=	half the carbonate ions after Phenolphthalein end point
2B	**=**	**all carbonate ions**
A-B	**=**	**all hydroxide ions**

Procedure:

Titration I : Standardization of HCl

20 ml of 0.1 N Na_2CO_3 solution is pipetted into a clean conical flask and two or three drops of methyl orange indicator is added to the solution. The solution is then titrated against hydrochloric acid taken in the burette. The end point is change of color from yellow to orange. The titrations are repeated to get concordant value.

Titration II: Estimation of Na_2CO_3 and NaOH in a given mixture

The given unknown solution is made upto 100 ml in a standard flask using distilled water. 20 ml of this made up solution is pipetted into a clean conical flask. Add 2 to 3 drops of phenolphthalein indicator and titrate against standard HCl. The disappearance of pink color is observed as end point and note down the titre value. Let it be A ml. To the same solution, add 2 to 3 drops of methyl orange indicator and continue the titration till the color changes from yellow to orange and note down the titre value after the phenolphthalein end point. Let it be B ml. The titration is repeated to get the concordant values of both A and B.

Titration 1 : Standardization of HCl

S.no	Volume of sodium carbonate soln. (ml)	Burette reading		Concordant Value	Indicator
		Initial	Final		Methyl orange

CALCULATION:

Volume of HCl $\quad\quad\quad\quad$ = $\quad\quad$ V_1 ml $\quad$ (end point)

Normality of HCl $\quad\quad\quad$ = $\quad\quad$? N_1

Volume of Na_2CO_3 (V_2) $\quad$ = $\quad\quad$ 20 ml

Normality of Na_2CO_3 (N_2) $\quad$ = $\quad\quad$ 0.1N

Normality of HCl $\quad$ (N_1) $\quad$ = $\quad$ (20 X 0.1) / V_1

$\quad\quad\quad\quad\quad\quad\quad\quad\quad\quad$ = $\quad\quad\quad$ _____ N

Titration II: Estimation of Na_2CO_3 and NaOH in a given mixture.

S.No	Volume of the unknown solution (ml)	Burette Reading (ml)		
		Initial	Volume consumed for Phenolphthalein end point (A)	Volume consumed for methyl orange end point after phph end point (B)
1				
2				
3				
Concordant Value			(A)	(B)

CALCULATION:

I Estimation of the amount of Na_2CO_3

Volume of HCl V_1 $\quad\quad\quad$ = $\quad\quad$ 2B (B is the titre value, after Phenolpthalein end point)

Normality of HCl $\quad\quad\quad$ = $\quad\quad$ N_1 (from Titration I)

Volume of mixture V_2 $\quad\quad$ = $\quad\quad$ 20ml

Normality of mixture N_2 $\quad$ = $\quad\quad$ 2B x N_1 / 20

Amount of Na_2CO_3 presents in whole the given solution $= N_2 \times 50 \times 1000*$

Equivalent weight of $CaCO_3$ is 50
* To convert g/L to mg/L

$$= \underline{\hspace{2cm}} \text{ mg / L or ppm}$$

II Estimation of amount of NaOH:

Volume of HCl V_1 $=$ (A-B) ml

Normality of HCl (N_1) $=$ N_1 (from Tit I)

Volume of mixture V_2 $=$ 20ml

Normality of Mixture N_2 $=$ (A-B) x N_1/ 20

Amount of NaOH presents in whole the given solution $= N_2 \times 50 \times 1000*$

Equivalent weight of $CaCO_3$ is 50
* To convert g/L to mg/L

$$= \underline{\hspace{2cm}} \text{ mg / L or ppm}$$

RESULT:

Amount of Na_2CO_3 present in the given solution = ----- ppm

Amount of NaOH present in the given solution   = ----- ppm

2. CONDUCTOMETRIC TITRATIONS – DETERMINATION OF STRENGTH OF HCl (Strong acid Vs Strong base)

EXERCISE NO.   **DATE**

AIM

To determine the strength of a given solution of HCl by conductometric titration with a given NaOH solution.

APPARATUS REQUIRED

Conductivity meter, conductivity cell, glass rod, beakers, burette, pipette, standard flask.

REAGENTS REQUIRED

HCl, NaOH, Conductivity Water.

PRINCIPLE

This principle is based on the measurement of the change of conductance with the help of the conductivity meter. The conductance of the solution depends on the number of ions (which are the actual carriers of current) and their ionic mobility.

During acid – base titration the base is added to the strong acid, H^+ ions are replaced by slow moving Na^+ ions. So the conductance of the solution decreases. After the neutralization point, further addition of excess alkali introduces fast moving OH^- ions and hence the conductance increases. The end point of the titration is determined graphically by plotting the conductance of HCl against the volume of alkali added. The point of intersection of the straight lines gives the end point at the volume axis (X-axis).

PROCEDURE

Make up the given HCl solution to 100 ml in a standard flask. Pipette out 10ml of the **made up HCl** in to a beaker. Dilute the solution with distilled water, so that the conductivity cell can be immersed well in the solution (100 ml). Stir the solution well with the help of a glass rod. Note down the conductance of the solution from the meter.

Fill the burette with standard NaOH solution and run down into the beaker in small increments [1 (or) 2ml] with gentle stirring of the contents of the beaker

Table 1
Standard NaOH Vs Unknown HCl (Pilot titration)

S.No.	Volume of NaOH (ml) added	Conductance in ohm^{-1}
1.		
2.		
3.		
4.		
5.		
6.		
7.		
8.		
9.		
10.		
11.		
12.		
13.		
14.		
15.		
16.		
17.		
18.		
19.		
20.		

Y
Conductance

Volume of NaOH

After each addition, stir the contents of the beaker and after an equilibrium time of 2-3 minutes, note the corresponding conductance value and tabulate it. Continue the titration till atleast 10 increments, after the conductance reaches a minimum and starts increasing. After the completion of titration, wash the conductance cell with distilled water and immersed in water.

Plot a graph between conductivity against volume of NaOH added. The intersection of two lines to the volume axis gives the end point.

Volume of NaOH required for neutralization is taken from graph (Fair) titration intersection point which is corresponding to the volume axis (X). In order to get accurate results, perform a fair titration, by adding NaOH in small increments near and beyond the end point. Calculate the strength of the given strong acid from the given NaOH can be calculated

Table 2
Standard NaOH Vs unknown HCl (Fair Titration)

S.No.	Volume of NaOH (ml) added	Conductance in ohm^{-1}
1.		
2.		
3.		
4.		
5.		
6.		
7.		
8.		
9.		
10.		
11.		
12.		
13.		
14.		
15.		
16.		
17.		
18.		
19.		
20.		

CALCULATION

Strength of Hydrochloric acid :

Volume of HCl (V_1)   =   10 ml

Normality of HCl (N_1)   = ... ?

Volume of NaOH(V_2)   = ... ml (obtained from graph)

Normality of NaOH(N_2)   = ...0.1 N

The strength of the Hydrochloric acid= -----N

RESULT

(i) The strength of the given HCl solution   = -----N.

Points for evaluation of exercise	Marks
Observation –Pilot &Fair titration	
Calculation	
Graph	
% of error	
Signature of the teacher with date	

3. ESTIMATION OF TOTAL HARDNESS, PERMANENT AND TEMPORARY HARDNESS BY EDTA METHOD

EXERCISE NO.

DATE

AIM

To estimate the amount of total hardness, permanent hardness and temporary hardness of a given sample of water by EDTA Method using ammonia buffer (pH=10) and eriochrome black – T indicator.

APPARATUS REQUIRED

Burette, Pipette, Conical Flask, Standard Volumetric flask, Funnel, Burner, Beaker 250ml

REAGENTS REQUIRED

EDTA Solution, Standard hard water, Sample water, Eriochrome black-T indicator (EBT), NH_3 - NH_4Cl buffer solution (pH10).

PRINCIPLE

Disodium Salt of ethylene diamine tetra acetic acid (EDTA),is used to determine the total hardness of the given hard water. The hardness causing metal ions (i.e. calcium and magnesium) form a wine-red coloured weak complex with Eriochrome Black - T indicator in the presence of a buffer solution. When EDTA is added, the indicator is replaced by EDTA and a stable complex is formed. Due to the liberation of Eriochrome Black - T indicator, wine red colour changes to steel blue. This is the end point for the titration between EDTA and hard water.

Ethylene diamine tetra acetic acid is a tetra carboxylic acid which has the following formula:

$$\begin{array}{cc} \text{HOOC H}_2\text{C} & \text{CH}_2\text{COOH} \\ & \text{N-CH}_2\text{-CH}_2\text{-N} \\ \text{HOOC H}_2\text{C} & \text{CH}_2\text{COOH} \end{array}$$

23
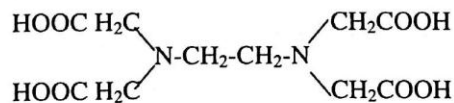
The entire reaction between Ca,. Mg ions and EB-T is represented as follows.

$$\begin{bmatrix} Ca^{2+} \\ Mg^{2+} \end{bmatrix} + EBT \rightarrow \begin{bmatrix} Ca^{2+} \\ \quad - EBT \\ Mg^{2+} \end{bmatrix} \text{Wine red (unstable)}$$

(Hard water sample)

$$\begin{bmatrix} Ca^{2+} \\ \quad EBT \\ Mg^{2+} \end{bmatrix} + EDTA \rightarrow \begin{bmatrix} Ca^{2+} \\ \quad EDTA \\ Mg^{2+} \end{bmatrix} + EBT \text{ (Blue)}$$

Stable colourless complex
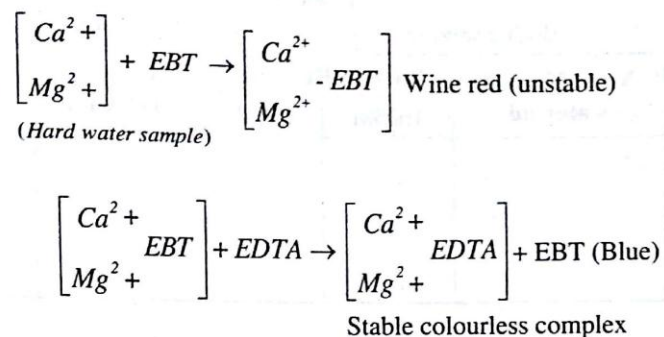
When the sample water is boiled, bicarbonates of calcium and magnesium are converted into carbonates and hydroxides, which can be removed by filtration.

The permanent hardness which is not removed by boiling is once again estimated by EDTA using Eriochrome Black-T-Indicator.

PROCEDURE

Standardization of EDTA

Pipette out 20 ml of standard hard water into a clean conical flask. Add 5ml of the buffer solution and 3 or 4 drops of the Eriochrome Black-T indicator. The solution turns wine red in color. Titrate the wine red coloured solution against EDTA taken in the burette. The change from wine red to steel blue colour is the end point. Repeat the titration for concordant values. Let the titer value be V_1 ml.

Determination of total Hardness

Pipette out 20 ml of the sample hard water into a clean conical flask. Add 5 ml of buffer solution and 3 or 4 drops of Eriochrome Black-T indicator. Titrate the wine red solution against EDTA. The change of wine red to steel blue is the end point. Repeat the titration for concordant values. Let V_2 be the volume of EDTA consumed.

Standardization of EDTA

Table 1
Std Hard water Vs EDTA solution

S.No.	Vol. of Std. hard water ml	Burette Reading		Vol. of EDTA ml	Indicator
		Initial	Final		
					EBT

Calculations:

1 ml of Standard hard water	=	1 mg of $CaCO_3$
Volume of standard hard water taken	=	20ml
20ml of Standard hard water	=	20mg of $CaCO_3$
Volume of EDTA consumed	=	V_1ml (from table-1)
V1ml EDTA solution	=	20mg $CaCO_3$

Therefore 1ml EDTA will be $\quad = \quad \dfrac{20}{V_1}$ mg of $equivalent$ $CaCO_3$

Determination of Total hardness

Table 2
Sample Hard water Vs EDTA Solution

S.No.	Vol. of Sample hard water ml	Burette Reading		Vol. of EDTA ml	Indicator
		Initial	Final		
					EBT

Volume of EDTA consumed $\quad = \quad V_2$ml (from table-2)

Now, if 1ml EDTA $\quad\quad = \quad \dfrac{20}{V_1} mg\ CaCO_3$

Then V_2ml EDTA $\quad\quad = \quad \dfrac{20}{V_1} x V_2\ mg\ CaCO_3$

$\quad\quad = \quad \underline{\quad\quad} mg\ CaCO_3$

If 20ml sample hard water taken for titration contains $\dfrac{20}{V_1} x V_2\ mg\ CaCO_3$,

Then, 1000ml will contain $\quad = \quad \dfrac{\left(\dfrac{20}{V_1}\right) x V_2}{20} x 1000 mg\ CaCO_3 \ = \ \dfrac{V_2}{V_1} x 1000 mg\ CaCO_3$

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

i.e Total hardness $\quad = \quad \underline{\quad\quad\quad} ppm$

Determination of Permanent hardness

Table 3
Boiled sample hard water Vs EDTA solution

S.No.	Vol. of boiled water ml	Burette Reading		Vol. of EDTA ml	Indicator
		Initial	Final		
					EBT

Volume of EDTA consumed $= V_3$ ml

If 1ml EDTA $= \dfrac{20}{V_1} mg\ CaCO_3$

Then, V_3ml EDTA $= \dfrac{20}{V_1} \times V_3 mg\ CaCO_3$

The boiled hard water sample is equivalent to permanent hardness $= \dfrac{20}{V_1} \times V_3\ mg\ CaCO_3$

Then, 1000ml will contain $= \dfrac{\left(\dfrac{20}{V_1}\right) \times V_3}{20} \times 1000 mg\ CaCO_3$

$= \dfrac{V_3}{V_1} \times 1000 mg\ CaCO_3$

$= \underline{\qquad} ppm.$

Permanent hardness $= \underline{\qquad} ppm.$

Estimation of Temporary hardness:

The temporary hardness of the given water sample = Total hardness – Permanent hardness

Determination of Permanent Hardness

Take 100ml of the hard water sample in a 250 ml beaker and boil gently for about one hour. Cool. Filter it into a 100 ml standard flask and make the volume upto the mark. Take 20 ml of this solution and proceed the titration in the same way. The volume of EDTA used corresponds to the permanent hardness of the water sample. Let the titer value be V_3 ml. Temporary hardness is calculated by subtracting permanent hardness from total hardness.

RESULT

The total hardness of sample hard water is =--------ppm

The permanent hardness of sample hard water is =--------ppm.

The temporary hardness of sample hard water is =--------ppm.

Points for evaluation of exercise	Marks
Observation – Standardization & Estimation	
Calculation	
% of error	
Signature of the teacher with date	

4 . ESTIMATION OF IRON BY POTENTIOMETRIC TITRATION

EXERCISE NO.   **DATE**

AIM

To estimate the amount of Fe^{2+} ion present in the given solution.

APPARATUSREQUIRED

Potentiometer assembly, 25ml burette, 10 ml pipette, 250ml beakers, standard flask, calomel and platinum electrodes.

REAGENTS REQUIRED

Ferrous ammonium sulphate, dil. H_2SO_4, Std $K_2Cr_2O_7$

PRINCIPLE

Measurement of Emf of a electrochemical cell using potentiometer and change in Emf due to the chemical (redox) reaction is monitored. In this potentiometric titration setup an indicator electrode (Pt electrode) and reference electrode (calomel) is coupled to form electrochemical cell for Fe^{2+} to Fe^{3+}.

Fe^{2+} is oxidizing to Fe^{3+} as $K_2Cr_2O_7$ as progressively added. Platinum electrode which is kept in contact with a mixture of Fe^{2+} and Fe^{3+} ions act as a redox electrode (indicating the redox reaction). The reduction potential of this single electrode depends on the ratio of $[Fe^{2+}/ Fe^{3+}]$ initially. During the titration of Fe^{2+} in H_2SO_4 medium with $K_2Cr_2O_7$, this ratio varies to a little extent at the beginning and suddenly near the end point. After the end point it the ratio changes very little. It can be noted that there is a sudden change in the ratio of $[Fe^{3+}/ Fe^{2+}]$ as the equivalence point is reached. This causes a sudden increase in the EMF of the cell at equivalence point.

Table 1
FAS Vs K$_2$Cr$_2$O$_7$ (Pilot Titration)

S. No.	Vol. of K$_2$Cr$_2$O$_7$ (ml)	EMF (volts)	ΔE (volts)
1.	0		
2.	1		
3.	2		
4.			
5.			
6.			
7.			
8.			
9.			
10.			
11.			
12.			
13.			
14.			
15.			
16.			
17.			
18.			
19.			
20.			

The cell set up: $Hg \mid HgCl_2$ (s), $\quad$ KCl (1N) $\parallel Fe^{2+} \mid Fe^{3+}$, Pt

The chemical reaction: $6Fe^{2+} + Cr_2O_7^{2-} + 14H^+ \rightleftharpoons 6Fe^{3+} + 2Cr^{3+} + 7H_2O$
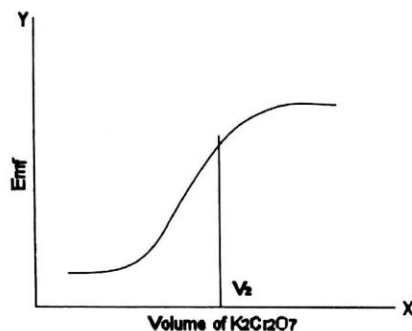
Pilot titration $\qquad\qquad$ **Fair Titration**
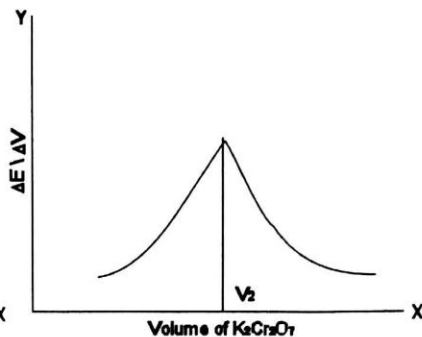
PROCEDURE:

i. The given ferrous iron solution is **made up to a known volume** (say 100ml) in a SMF following the standard procedure with usual precautions.

ii. Exactly 10ml of the **made up Fe^{2+} solution** is pipette out into a clean 100ml beaker. About 10ml of dil. H_2SO_4 and 100ml of distilled water are added to it.

iii. A platinum electrode is dipped into this solution and coupled with the standard calomel electrode. The resultant cell is then incorporated into the potentiometric circuit.

iv. Standard $K_2Cr_2O_7$ solution, which is taken in a burette, is added in installments of 1ml into the beaker and the cell e.m.f is measured after each addition by proper mixing.

v. The process is continued till and also well beyond the neutralization point as indicated by an abrupt change in the e.m.f

Table 2

FAS Vs K$_2$Cr$_2$O$_7$ (Fair Titration)

S. No.	Vol. of K$_2$Cr$_2$O$_7$ (ml)	EMF (volts)	ΔE (volts)	ΔV (ml)	$\Delta E/\Delta V$ (volts/ml)
1.					
2.					
3.					
4.					
5.					
6.					
7.					
8.					
9.					
10.					
11.					
12.					
13.					
14.					
15.					
16.					
17.					
18.					
19.					
20.					

CALCULATION

Volume of pipette solution (FAS) $=$ V_1 ml

Volume of $K_2 Cr_2 O_7$ $=$ V_2 ml (from graph)

Normality of $K_2 Cr_2 O_7$ $=$ N_2

Normality of FAS (N_1) $=$?

Strength of FAS $= N_1$ $=$ $\dfrac{V_2 N_2}{V_1}$

Amount Fe^{+2} (g/lit) $=$ Eq. wt x Normality of Fe^{+2}

Amount of Fe^{+2} in 100ml $=$ $\dfrac{Normality\ of\ Fe^{+2}\ x\,55.85}{10}$

i. Note the volume $K_2Cr_2O_7$ solution required for complete oxidation of Fe^{2+} solution from the plot of e.m.f versus the volume of the standard $K_2Cr_2O_7$ solution added. The range at which the endpoint lies may be evaluated

ii. Calculate the normality of given Fe^{2+} solution using the formula $N_1V_1 = N_2V_2$

iii. One more similar titration is performed by adding 0.1ml portions of standard $K_2Cr_2O_7$ solution close to the end point (1ml on either side of the range) and tabulating the measured e.m.f corresponding to each addition.

iv. Graph is plotted(i) Emf vs Volume of $K_2Cr_2O_7$ addition

(ii) $\Delta E/\Delta V$ vs Volume of $K_2Cr_2O_7$ addition

v.The exact end point can be determined from the plot of $\Delta E/\Delta V$ versus volume of standard $K_2Cr_2O_7$ solution

RESULT

The weight of Iron present in the given solution   =-------g

Points for evaluation of exercise	Marks
Observation –Pilot & Fair titration	
Calculation	
Graph	
% of error	
Signature of the teacher with date	

## Ex. No. 5 Estimation of amount of Chloride of a water sample.

AIM:

To estimate the amount of chloride in a water sample by Mohr's method.

PRINCIPLE:

It is an example of precipitation reaction. The reaction between chloride and silver nitrate is direct and simple. It proceeds as follows:

$$AgNO_3 + NaCl \quad \rightarrow \quad AgCl + NaNO_3$$
$$Ag^+ + Cl^- \quad\quad \rightarrow \quad\quad AgCl$$
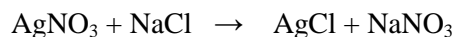
The completion of the reaction in this case is observed by employing potassium chromate (K_2CrO_4) solution as the indicator. At the end point, the yellow colour changes into reddish brown due to the reaction

$$2AgNO_3 + K_2CrO_4 \rightarrow Ag_2CrO_4 + 2KNO_3$$
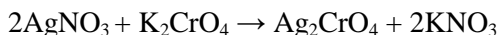
K_2CrO_4 indicator will not be precipitated as Ag_2CrO_4 until all the chlorides in the solution have been precipitated as AgCl.

PROCEDURE:

Titration I - Standardization of silver nitrate solution

20ml of standard NaCl solution (N/20) is pipetted out into a clean conical flask. 1ml of 2% K_2CrO_4 indicator is added to it. The solution turns yellow in color. It is titrated against $AgNO_3$ solution taken in the burette. During each addition of $AgNO_3$, the content in the conical flask is shaken well. At the end point, the yellow colour changes to brownish red colour. The titration is repeated for concordant values.

Titration II: Estimation of Chloride

The given chloride solution is made up to 100 ml in a standard flask. Exactly 20ml of this solution is pipetted out into a clean conical flask. To this solution 1.0 ml of 2% K_2CrO_4 indicator is added. It is titrated against standardized $AgNO_3$ solution from the burette. The addition of $AgNO_3$ solution is continued until the solution produced a reddish brown tinge. The titration is repeated for concordance. From the volume of $AgNO_3$ consumed, the strength of chloride and hence its amount is calculated.

Table 1. Standardization of AgNO₃.

S.No	Volume of NaCl solution (ml)	Burette Reading (ml)		Concordant Value (ml)	Indicator
		Initial	Final		
					Potassium chromate

Table 2. Estimation of Chloride

S.No	Volume of chloride solution (ml)	Burette Reading (ml)		Concordant Value (ml)	Indicator
		Initial	Final		
					Potassium chromate

CALCULATION

Titration I - Standardization of AgNO₃ Solution

Normality of NaCl Solution	=	N_1
Volume of NaCl Solution	=	20ml (V_1)
Volume of AgNO₃ Solution	=	V_2 (end point)
Normality of AgNO₃ Solution	=	___ N_2

$$N_2 = V_1 \times N_1 / V_2$$

Titration II - Estimation of Chloride

Volume of Chloride solution	=	20ml (V_1)
Normality of Chloride solution	=	____ N_1
Volume of AgNO₃ Solution	=	V_2 (end point)
Normality of AgNO₃ Solution	=	N_2 (from tit I)

$$N_1 = V_2 \times N_2 / V_1$$

Amount of chloride present in the whole of the given solution = $(N_1 \times 35.46) / 10$

= ----- g/ 100 mL

RESULT:

Amount of chloride present in the whole of the given solution is ------- g / 100 mL.

6 DETERMINATION OF MOLECULAR WEIGHT OF A POLYMER BY VISCOSITY AVERAGE METHOD

EXERCISE NO.   **DATE**

AIM

To determine the molecular weight of a polymer in solution by using a viscometer.

APPARATUS REQUIRED

Ostwald's viscometer, Volumetric flask, stop watch, standard flasks

REAGENTS REQUIRED

Polymer, suitable solvents

PRINCIPLE

If a polymer is soluble in a suitable solvent, measurement of solution viscosity provides a simple and convenient method for molecular weight determination. In a capillary viscometer (Ostwald/Ubbelhode) the viscosity of a liquid is proportional to the time taken by a known volume of liquid to flow through a capillary under a specified hydrostatic pressure at a fixed temperature. The conditions for flow should ensure that the flow is laminar. Using Poiseuille's equation it is possible to show that if t, η and ρ are the flow time, viscosity and density of a solution respectively; and t_0, η_0 and ρ_0 are those of the pure solvent, then

$$\frac{\eta}{\eta_0} = \frac{\rho}{\rho_0} \cdot \frac{t}{t_0}$$

The value of $\frac{\eta}{\eta_0}$, is known as the relative viscosity η_{rel}. In dilute solutions, which are often employed for molecular weight determinations, ρ is not much different from ρ_0 and hence

$$\eta_{rel} = \frac{\eta}{\eta_0} = \frac{t}{t_0}$$

The specific viscosity η_{sp} is definded as

$$\eta_{sp} = \eta_{rel} - 1$$

A plot of $\dfrac{\eta_{sp}}{C}$ vs C is a straight line for dilute solutions, the intercept

$$\frac{\lim \eta_{sp}}{C \to 0 \; C} = \eta_{int}$$

of which is known as the intrinsic viscosity η_{int}.

The Staudiger - Mark-Houwink equation which relates η_{int} with molecular weight

$$\eta_{int} = K(M)^{\alpha}$$

where 'K' is an empirical parameter characteristic of a particular solute-solvent pair and 'α' is a 'shape' parameter, which can vary from about 0.5 for well-coiled polymers in poor solvents to about 2 for rigidly extended 'rod' like polymers. From known values of K and α, Molecular weight can be determined.
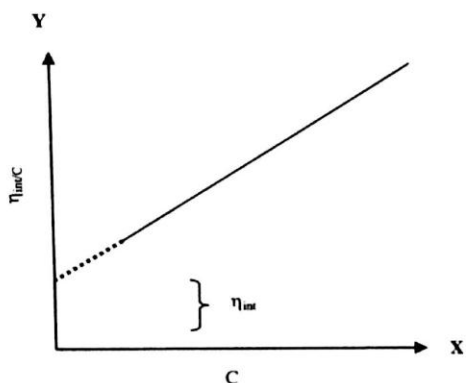
Plot of η_{sp}/C Vs Concentration of polymer solution to find out intrinsic viscosity

S.No.	Concentration of the polymer solution	Time of flow in sec (average)	Relative viscosity $\eta/\eta_0 = t_s/t_0$	Specific viscosity $\eta_{sp} = \eta/\eta_0 - 1$	Reduced viscosity η_{sp}/C
1.	Pure Solvent	t_0			
2.	0.1%	t_s			
3.	0.2%	t_s			
4.	0.3%	t_s			
5.	0.4%	t_s			
6.	0.5%	t_s			

CALCULATION

Solvent used Water

$$\eta_{int} = K \times M^\alpha$$

$$\log \eta_{int} = \log K + \alpha \log M$$

$$\alpha \log M = \log \eta_{int} - \log K$$

$$\log M = \frac{\log \eta_{int} - \log K}{\alpha}$$

$$M = Anti \log \frac{[\log \eta_{int} - \log K]}{\alpha}$$

Plot η_{sp}/C Vs C.

Extrapolate the straight line to Y axis

The intercept is η_{int}.

From the relationship, $\eta_{int} = KM^\alpha$ where K and α are constants,

(For PVA solution $K = 45.3 \times 10^{-3}$, $\alpha = 0.64$)

PROCEDURE

Preparation of various concentrations of polymer in water (solvent):

1% solution of polymer in water will be supplied. We need to prepare at least '5' dilutions viz 0.1%, 0.2%, 0.3%, 0.4% and 0.5% polymer in water before carrying out the experiment

Dilutions can be done by using volumetric expression,

$$V_1 N_1 = V_2 N_2$$

Eg: To prepare 100ml of 0.2% diluted solution from a 1% solution, volume is

$$V_1 = \frac{V_2 \times N_2}{N_1} = \frac{100ml \times 0.2\%}{1\%} = 20ml$$

Similarly, any other dilutions can be prepared by the above method.

Set up the Ostwald (or Ubbelhode) viscometer and measure the flow time (t_0) of a fixed volume of the pure solvent. Take an average of three readings. Rinse the viscometer thoroughly with the most dilute solution, measure the flow time (t_1) keeping the flow-volume the same. Repeat the procedure for other solutions.

Calculate η_{rel} and η_{sp}. Plot η_{sp}/C vs C, extrapolate to C=0 to obtain η_{int}. From the given values of K and α, calculate the molecular weight.

RESULT

Volume of polymer liquid to be used for each measurement = _____ml

The molecular weight of the given polymer is ---------.

| Points for evaluation of exercise	Marks
Observation	
Calculation	
Graph	
% of error	
Signature of the teacher with date	

Ex. No. 7 DETERMINATION OF THE STRENGTH OF A MIXTURE OF ACETIC ACID AND HYDROCHLORIC ACID BY CONDUCTOMETRY

Aim:

To estimate the strength of the mixture of acetic acid and hydrochloric acid present in a given mixture by conductometrically.

Principle:

The conductivity of the solution is related to the mobility of ions which in turn related with the size of the ions. When a mixture of acids like a strong acid (HCl) and a weak acid (acetic acid) are titrated against a strong base (NaOH), strong acid reacts first followed by a weak acid. When the titration of strong acid and strong base are carried out, there is a decrease in conductivity as highly mobilized hydrogen ions are replaced by sodium ions.

$$NaOH + HCl \longrightarrow NaCl + H_2O$$
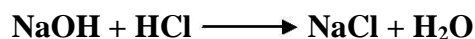
When the whole strong acid is consumed, base reacts with weak acid and conductivity increases as unionised weak acid becomes the ionised salt.

$$CH_3COOH + Na^+ + OH^- \longrightarrow CH3COO^- + Na^+ + H_2O$$
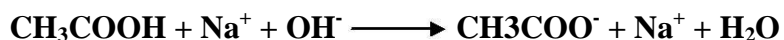
After both the acids are consumed, there is a steep increase in conductivity which gives the end point and this increase in conductivity is due to the fast moving hydroxyl ions from the base. From this, amount of base consumed for acid and in turn, the amount of acids present is calculated.

PROCEDURE:

The given mixture of acids is made up to 100 ml using distilled water. 10 ml of this made up solution is pipetted out into clean beaker and 100 ml of distilled water is added. The conductivity cell is dipped into the test solution and titrated against NaOH (0.5 ml) interval with proper stirring. The conductance is measured after each 0.5 ml addition of NaOH at various stages of neutralization. After complete neutralization, the amount of acid present in the given mixture is determined based on the volume of NaOH consumed. Volume of base consumed for strong acid and weak acid are determined by plotting a graph between conductance and volume of base added, where first end point corresponds to strong acid and second end point corresponds to weak acid.
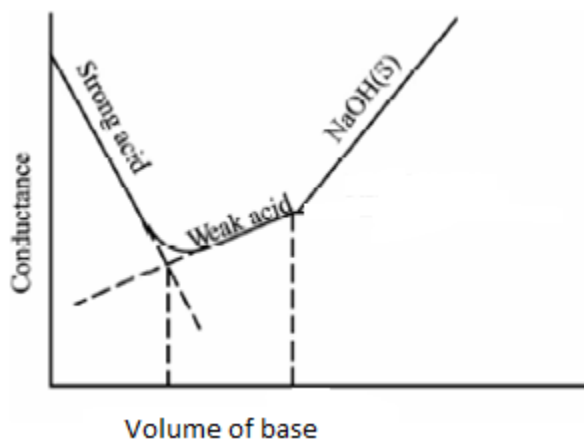
Volume of base

TABLE-1: Titration between mixture of acids and NaOH

S.No	Volume of NaOH added (ml)	Conductance (ohm^{-1})
1		
2		
.		
.		
.		
30		

CALCULATION

Volume of mixture	= 10ml
Normality of HCl	= ----N
Volume of NaOH	= V_1 ml [I^{st} end point from graph]
Normality of NaOH	= 0.1 N
Strength of HCl	= $\dfrac{V_1 \times 0.1}{10}$
	= ----------N.

Volume of mixture	$= 10ml$
Normality of CH_3COOH	$= $ -----N
Volume of NaOH	$= V_2 - V_1 ml$ [V_2-2nd end point from graph]
Normality of NaOH	$= 0.1 N$
Strength of CH_3COOH	$= \dfrac{0.1 \times (V_2 - V_1)}{10}$
	$= $ -----------N

RESULT:

1. The strength of HCl present in the whole of the given solution _____ N.

2. The strength of CH_3COOH present in the whole of the given solution _____ N.

Ex. No. 8 DETERMINATION OF STRENGTH OF AN ACID USING pH METER

Aim

　　To find out the strength of given hydrochloric acid solution by titrating it against sodium hydroxide (0.1N) using pH meter.

Principle

　　When an alkali is added to an acid solution, the pH of the solution increases slowly, but at vicinity of the end point, the rate of change of pH of the solution is very rapid. From the sharp break in the curve, we can find out the end point, from which the strength of HCl can be calculated.

Procedure

　　First standardize the pH meter using different buffers of known pH, then wash the glass electrode and reference electrode with distilled water and then with the acid solution. The given acid is made up to 100 ml using distilled water. 10 ml of this made up solution is pipetted out into a 250 mL clean beaker and 100 ml of distilled water is added, so that the glass electrode as well as the reference electrode is completely dipped. Note the pH of the pure acid solution. Fill the burette with standard NaOH solution and run down into the beaker in small increments (1mL). Stir the solution well using glass rod. Note down the pH of every successive addition. Continue the titration till beyond the neutralization point as indicated by an abrupt change in pH. (atleast 30 increments). Plot a graph between volume of NaOH against pH. The midpoint of the 'S' shaped curve of the graph gives the equivalence point. Near the end point add very small amount of NaOH, because change in pH will be very much appreciable when the acid is neutralized, further addition of such a small quantity of 0.01 mL raises the pH about 9 to 10.
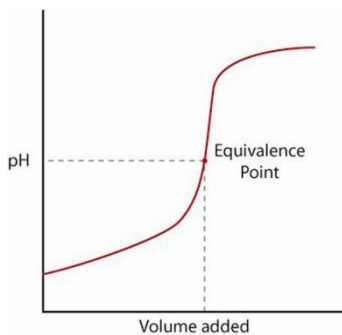
In order to get an accurate end point, perform one more similar titration by adding 0.2 mL of standard NaOH solution close to the end point (1.0 mL on either side of the range) and measure the pH of every addition. Plot a fair graph between volume of NaOH against Δ pH / ΔV. Find out the exact end point from the fair graph. The peak point of the curve from the fair graph gives the equivalence point.
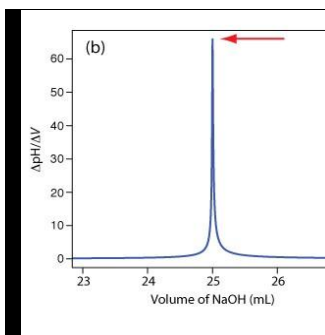
Table 1: Pilot Titration between HCl Vs NaOH.

S.No	Volume of NaOH (V) ml	pH
1.		
2.		
3.		
….		
30.		

Table 2: Fair Titration between HCl Vs NaOH.

S.No	Volume of NaOH (V) ml	pH	Δ pH	ΔV	Δ pH / ΔV
1.					
2.					
3.					
….					
20.					

Pilot Graph

Fair Graph

CALCULATION

Volume of NaOH V_1 = ------------- ml [from FAIR graph]

Strength of NaOH N_1 = ------------ N

Volume of HCl V_2 = ------------- ml

Strength of HCl N_2 =   N

Therefore strength of HCl (N_2) $= V_1 \times N_1 / V_2$

= -----------N.

RESULT:

The strength of given hydrochloric acid solution is ------- N.