

Experiment - I

Aim:

To determine the amount of sodium carbonate (Na_2CO_3) and sodium hydroxide (NaOH) in a mixture using hydrochloric acid

Apparatus required

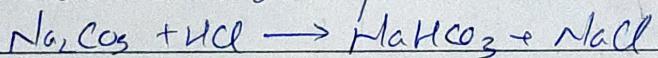
Conical flask, 10ml standard flask, 20 ml pipette, burette, funnel, glass rod.

Chemical Required

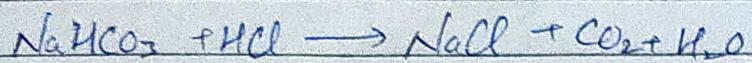
Sodium carbonate (Na_2CO_3), Sodium hydroxide (NaOH), hydrochloric acid (HCl), Phenolphthalein, Methyl orange, Distilled water

Principle

The titration of a mixture of NaOH and Na_2CO_3 with a standardized HCl solution has two equivalence points. The first equivalence point is due to the conversion of all the carbonate to bicarbonate and sodium hydroxide to sodium chloride as follows:



The pH of the resulting solution is around therefore phenolphthalein could be used as an indicator. The second equivalence point is due to the reaction of the resulting bicarbonate with an excess of HCl solution as follows:



In this stage methyl orange is used to find the equivalence point because the pH of the solution at this point will be around 3.8

Result

Normality of the given HCl solution = 0.05 N

Amount of Na_2CO_3 present in the given solution = 0.2405 g

Amount of NaOH present in the given solution = 0.153 g

Table-I

Sl No.	Volume of pipette solution (ml)	Burette Reading (ml)		Concordant Value	Indicator
		Initial	Final		
1	20	0	19.6	19.6	Methyl orange
2	20	0	19.6		

Table-II

Sl No.	Volume of pipette solution (ml)	Burette Solution (ml)			Concordant Value	
		Initial	Vol. consu. Phenolphthalein end point (A)	Vol. consu. Methylorange end point (X)	Phenolphthalein	Methyl orange
1	20	0	23.9	32.8	23.9	32.8
2	20	0	23.9	32.8		

Phenolphthalein endpoint (concordant value) = A = 23.9 ml

Methyl orange endpoint (concordant value) = X = 32.8 ml.

Vol. consumed for methyl orange endpoint after Phenolphthalein end point B = X - A = 32.8 - 23.9 = 8.9 ml

Calculation

$$\text{Volume of HCl}(V_1) = 19.6 \text{ ml} \quad \text{Volume of Na}_2\text{CO}_3(V_2) = 20 \text{ ml}$$

$$\text{Normality of Na}_2\text{CO}_3(N_2) = 0.051 N$$

$$\begin{aligned}\text{Normality of HCl (N}_1) &= (20 \times 0.05)/19.6 \\ &= 0.051 N\end{aligned}$$

Estimation of the amount of Na_2CO_3

$$\text{Volume of HCl (V}_1) = 17.8$$

$$\text{Normality of HCl} = 0.051 N$$

$$\text{Volume of mixture} = 20 \text{ ml}$$

$$\text{Normality of } \text{Na}_2\text{CO}_3(N_1) = (17.8 \times 0.051)/20 = 0.045 N$$

$$\text{Amount of Na}_2\text{CO}_3 \text{ present} = \frac{0.045 \times 53}{10} = 0.2405 \text{ g}$$

Estimation of the amount of NaOH

$$\text{Volume of HCl solution (V}_1) = 19.6 - 8.8 = 10.8 \text{ ml}$$

$$\text{Normality of HCl solution (N}_1) = 0.051 N$$

$$\text{Volume of mixture (V}_2) = 20 \text{ ml}$$

$$\text{Normality of NaOH} = (15 \times 0.051)/20 = 0.03825 N$$

$$\text{Amount of NaOH} = \frac{(0.03825) \times 40}{10} = 0.153 \text{ g}$$

Experiment - 2.

Aim

To estimate the amount of total hardness, permanent hardness and temporary hardness of a given water sample by EDTA method

Apparatus Required

Burette, pipette, conical flask, standard volumetric flask, funnel.

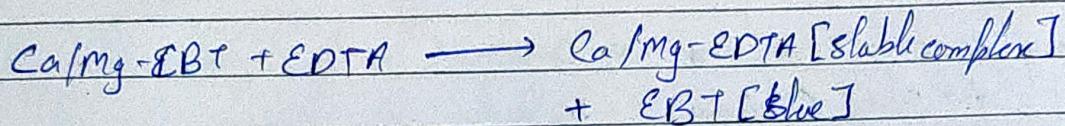
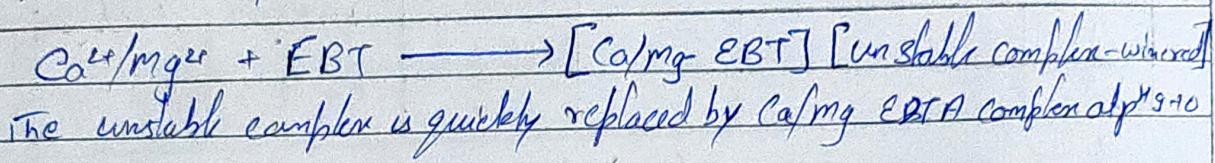
Reagents Required

Standard hard water, sample water, boiled water, Eriochrome black-T (EBT) indicator, ammonical buffer ($\text{NH}_3\text{-NH}_4\text{Cl}$) solution and ethylenediamine tetra acetic acid (EDTA).

Principle

Ethylenediamine tetra acetic acid disodium salt is used to determine the total hardness, permanent hardness (sulphate and chlorides of calcium & magnesium) and temporary hardness (bicarbonates of calcium & magnesium) of the given water sample. The hardness causing metal ions (Ca^{2+} & Mg^{2+}) form red wine coloured weak complex with Eriochrome black-T indicator in the presence of a buffer solution. The indicator is replaced by EDTA and a stable complex is formed with the addition of EDTA. Due to the removal of EBT indicator, wine-red colour changes to steel blue.

The entire reaction between metal ions and EBT
representable as follows:



Result

The total hardness of the given sample = 471.61 ppm

The permanent hardness of the given sample = 309.9 ppm

The temporary hardness of the given sample = 161.69 ppm

Table 1 - Standardisation of EDTA

S.No.	Volume of Sample Hardwater(mL)	Burrette Reading		Volume of EDTA solution	Indicator
		Initial	Final		
1	20	0	22.9	22.9	Eriochrome black-T
2	20	0	22.9		

1mL of standard hard water = 1mg of CaCO_3

Volume of standard hard water taken = 20mL

20mL of standard hard water taken = 20mg of CaCO_3

Volume of EDTA consumed = 22.9mL

22.9mL EDTA solution = 20mg of CaCO_3

Therefore 1mL EDTA will be = $\frac{20}{22.9} = 0.873 \text{ mg of } \text{CaCO}_3$

Table 2 (Determination of Total hardness)

S.No.	Volume of Sample Hardwater (mL)	Burrette Reading		Volume of EDTA solution(mL)	Indicator
		Initial	Final		
1	20	0	10.8	10.8	Eriochrome black-T
2	20	0	10.8		

Volume of EDTA consumed = 10.8 mL.

1mL of EDTA = 0.873 mg of CaCO_3

10.8mL of EDTA = $(0.873) \times 10.8 = 9.432 \text{ mg of } \text{CaCO}_3$

If 20mL of sample hard water taken for titration = $\frac{20}{22.9} \times 10.8 = 9.432$

Then, 1000mL will contain = $\left(\frac{20}{22.9} \times 10.8\right) \times \frac{1000}{20} = 471.61$

Table 3.

S.No	Volume of Sample Hard water(ml)	Burette Reading (ml)		Volume of EDTA solution (ml)	Indicator
		Initial	Final		
1	20	0	7.1	7.1	Eriochrome Black-T
2	20	0	7.01		

$$\text{Volume of EDTA consumed} = 7.1 \text{ mL}$$

$$\text{1 ml of EDTA} = 0.873 \text{ mg of CaCO}_3$$

$$7.1 \text{ mL of EDTA} = 6.198 \text{ mg of CaCO}_3$$

If 20 ml of sample hard water taken for titration = 6.198 mg $(\frac{20}{V_1} \times V_2)$

$$\text{Then, 1000 ml will contain} = \left(\frac{20}{V_1} \times V_2 \times 1000 \right) / 20 = 309.91 \text{ ppm}$$

$$\text{Permanent hardness} = 309.91 \text{ ppm}$$

$$\begin{aligned}\text{Temporary hardness} &= \text{Total hardness} - \text{Permanent hardness} \\ &= 471.60 \text{ ppm} - 309.9 \text{ ppm} \\ &= 161.69 \text{ ppm}\end{aligned}$$

Experiment - 3

Aim

To estimation the amount of chloride content in a water sample by Mohr's method

Apparatus required

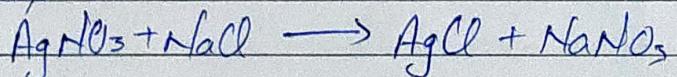
Burette, pipette, conical flask, glassrod, funnel, standard flask

Reagents required

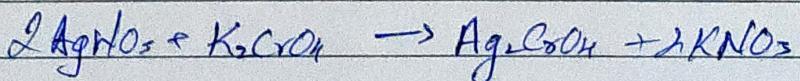
Silver nitrate solution, standard NaCl solution, K_2CrO_4 indicator, sample water and distilled water.

Principle

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



The completion of the reaction in this case is observed by employing K_2CrO_4 solution as the indicator. At the end point, the yellow colour of solution change into reddish brown due to the reaction



~~K₂CrO₄~~ indicator will not be precipitated as Ag₂CrO₄ until all the chlorides in the solution have been precipitated as AgCl.

Result

Amount of chloride present in the whole of the given solution
= 0.0439 g/l

Table -1 Densitration of silver nitrate solution

Sno.	Volume of pipette solution (ml)	Burrell reading		Concordant value(ml)	Indicator used
		Initial	Final		
1	10	0	13.4	13.4	K ₂ C ₂ O ₄
2	10	0	13.4		

$$\text{Normality of NaCl solution} = 0.017 N$$

$$\text{Volume of NaCl solution} = 10 \text{ ml}$$

$$\text{Volume of AgNO}_3 \text{ solution} = 13.4 \text{ ml}$$

$$\text{Normality of AgNO}_3 \text{ solution} = (N) \rightarrow \frac{0.017 \times 10}{13.4} = 0.0126 N$$

Table -2

SNo	Volume of pipette solution (ml)	Burrell reading		Concordant value(ml)	Indicator used
		Initial	Final		
1	10	0	13.2	13.2	K ₂ CrO ₄
2	10	0	12.7	12.7	

$$\text{Volume of chlorine solution} = 20 \text{ ml}$$

$$\text{Normality of AgNO}_3 = 0.0126 N$$

$$\text{Volume of AgNO}_3 = 19.7 \text{ ml}$$

$$\text{Normality of chlorine solution} \rightarrow \frac{0.0126 \times 19.7}{20} = 0.0124 N$$

Amount of chloride present

$$= \frac{0.0124 \times 35.45}{10} = 0.0439 \text{ g/l}$$

Experiment - 4

Aim

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

Apparatus Required

Burette, pipette, standard measuring flask, glass rod, Beaker, wash bottle, funnel, Conductivity meter, conductivity cell

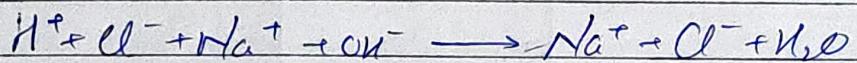
Chemical Required

NaOH solution (0.1 N), distilled water, HCl solution whose concentration is to be found.

Principle

The principle of the conductometric titration is that during a titration process, one ion is replaced with another and the difference in the ionic conductivities of these ions directly impacts the overall electrolyte conductivity of the solution. It is evident from the equation that as NaOH solution is gradually added, the H^+ ion having high ionic conductance are replaced by Na^+ having lower ionic conductance and hence the conductivity of the solution gradually decrease. At the equivalent point the conductivity would be minimum due to replacement of all H^+ ion by Na^+ ion.

After the equivalence point Na^+ and OH^- would accumulate in the solution and conductance of the solution will again



Result

The strength of the given HCl solution is 0.092 N

Observation table

NaOH vs HCl Titration

S.No	Vol. of NaOH added (ml)	Conductance (ohm^-1)
1	0	3.532
2	1	3.278
3	2	3.032
4	3	2.797
5	4	2.523
6	5	2.288
7	6	2.084
8	7	1.812
9	8	1.564
10	9	1.348
11	10	1.164
12	11	1.903
13	12	2.268
14	13	2.437
15	14	2.789
16	15	2.913

Fair titration

S.No	Vol. of NaOH added (ml)	Conductance (ohm^-1)
1	8.0	1.589
2	8.2	1.864
3	8.4	1.092
4	8.6	1.057
5	8.8	1.022
6	9.0	0.981
7	9.2	0.921
8	9.4	0.946
9	9.6	0.968
10	9.8	0.996
11	10	1.219
12	10.2	1.438
13	10.4	1.683
14	10.6	1.763
15	10.8	1.828
16	11	1.987

Calculation :-

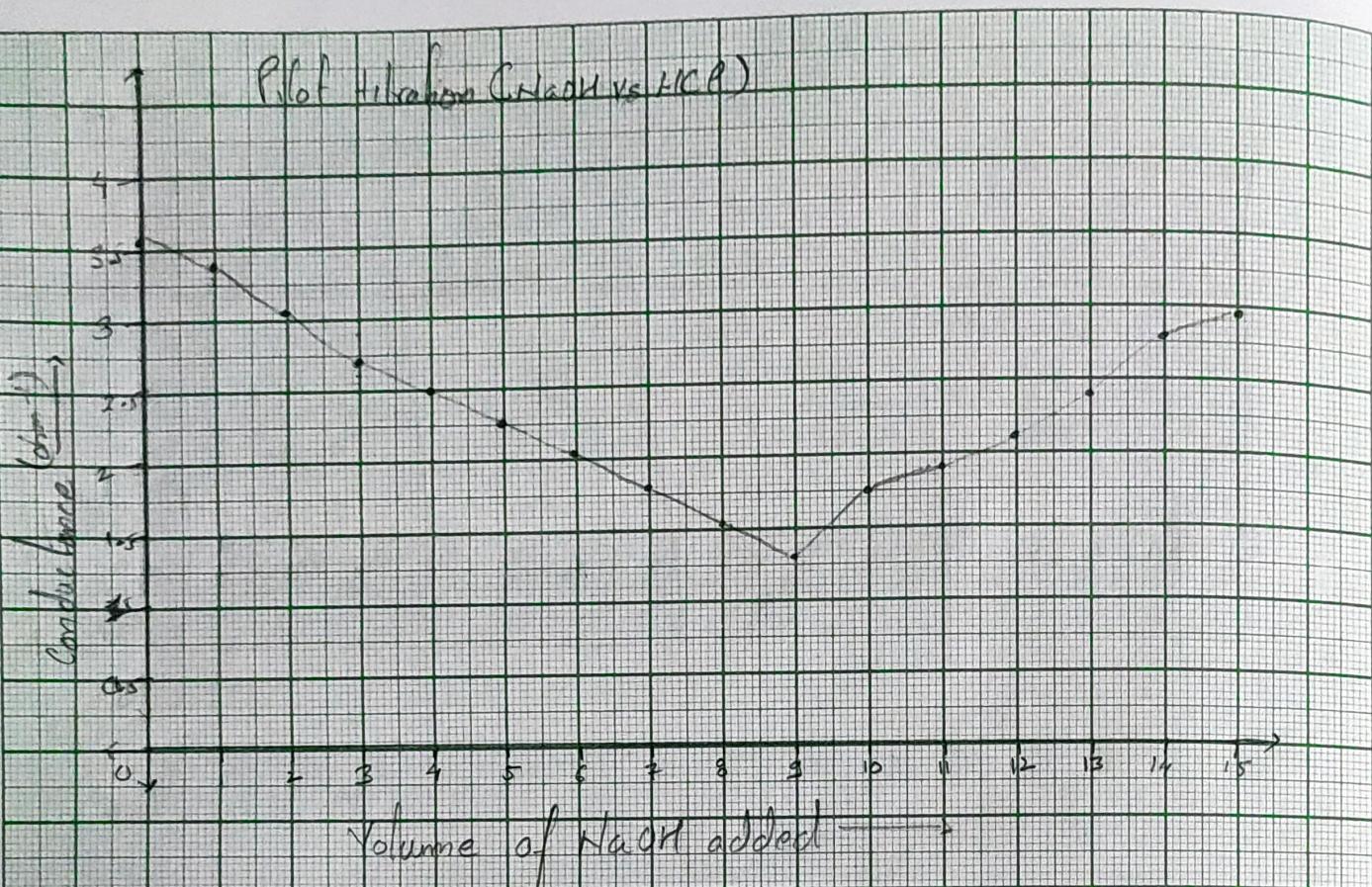
$$\text{Volume of HCl solution} = 10 \text{ mL}$$

$$\text{Volume of NaOH solution} = 9.2 \text{ mL (From graph)}$$

$$\text{Normality of NaOH} = 0.1 \text{ N}$$

$$\text{Normality of HCl} = \frac{0.1 \times 9.2}{10} = 0.092 \text{ N}$$

Pilot titration (NaOH vs HCl)



Fair titration (NaOH vs HCl)



Experiment - 5

Aim

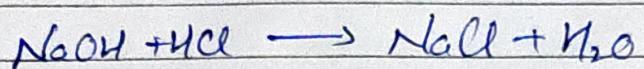
To estimate the strength of mixture of acetic acid and hydrochloric acid present in a given mixture using conductometric titration.

Apparatus and Reagents required

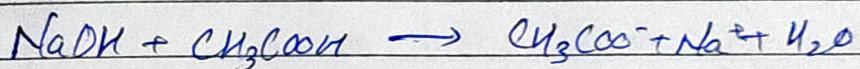
Conductivity meter, Conductivity cell (Platinum plates), Burette, pipette, Conical flask, standard measuring flask (SMP), NaOH , given unknown solution.

Principle

The principle of conductometric titration is based on the fact that during the titration, one of the ions is replaced by the other ions and invariably these two ions differ the ionic conductivity with the result that conductivity of the solution varies during the course of titrations. During the titration of strong base against a strong acid is carried out, there two break points observed in the graph. The first break point corresponds to the neutralisation of strong acid. After the complete neutralisation of strong acid, at the second break point the neutralisation weak acid starts and conductivity increases slightly as unionized weak acid becomes the ionized salt. Finally, the conductance increases due to the excess of strong base OH^- ions.



when the whole strong acid is consumed, base reacts with weak acid and



Result

The strength of HCl present in the whole of the solution = 0.03N

The strength of CH₃COOH present in the whole of the Solution = 0.0275N

Observation Table

SNO.	Volume of NaOH added (mL)	Conductance (ohm^{-1})
1	0	7.86
2	0.5	7.39
3	1	6.72
4	1.5	6.25
5	2	5.52
6	2.5	5.15
7	3	4.68
8	3.5	4.13
9	4	3.62
10	4.5	3.29
11	5	2.84
12	5.5	2.37
13	6	2.22
14	6.5	2.35
15	7	2.49
16	7.5	2.58
17	8	2.64
18	8.5	2.72
19	9	2.88
20	9.5	2.96
21	10	3.14
22	10.5	3.21
23	11	3.64
24	12.5	3.89
25	12	4.18
26	12.5	4.31
27	13	4.69
28	13.5	4.94
29	14	5.23
30	14.5	5.51
31	15	5.87
32	15.5	6.3
33	16	6.72
34	16.5	7.12
35	17	7.59
36	17.5	7.82
37	18	8.24

Strength of HCl

Volume of mixture = 20ml

Volume of NaOH (V_1) = 6ml (From graph)

Normality of NaOH = 0.1N

$$\text{Strength of HCl} = \frac{0.1 \times 0.1}{20} = 0.005N$$

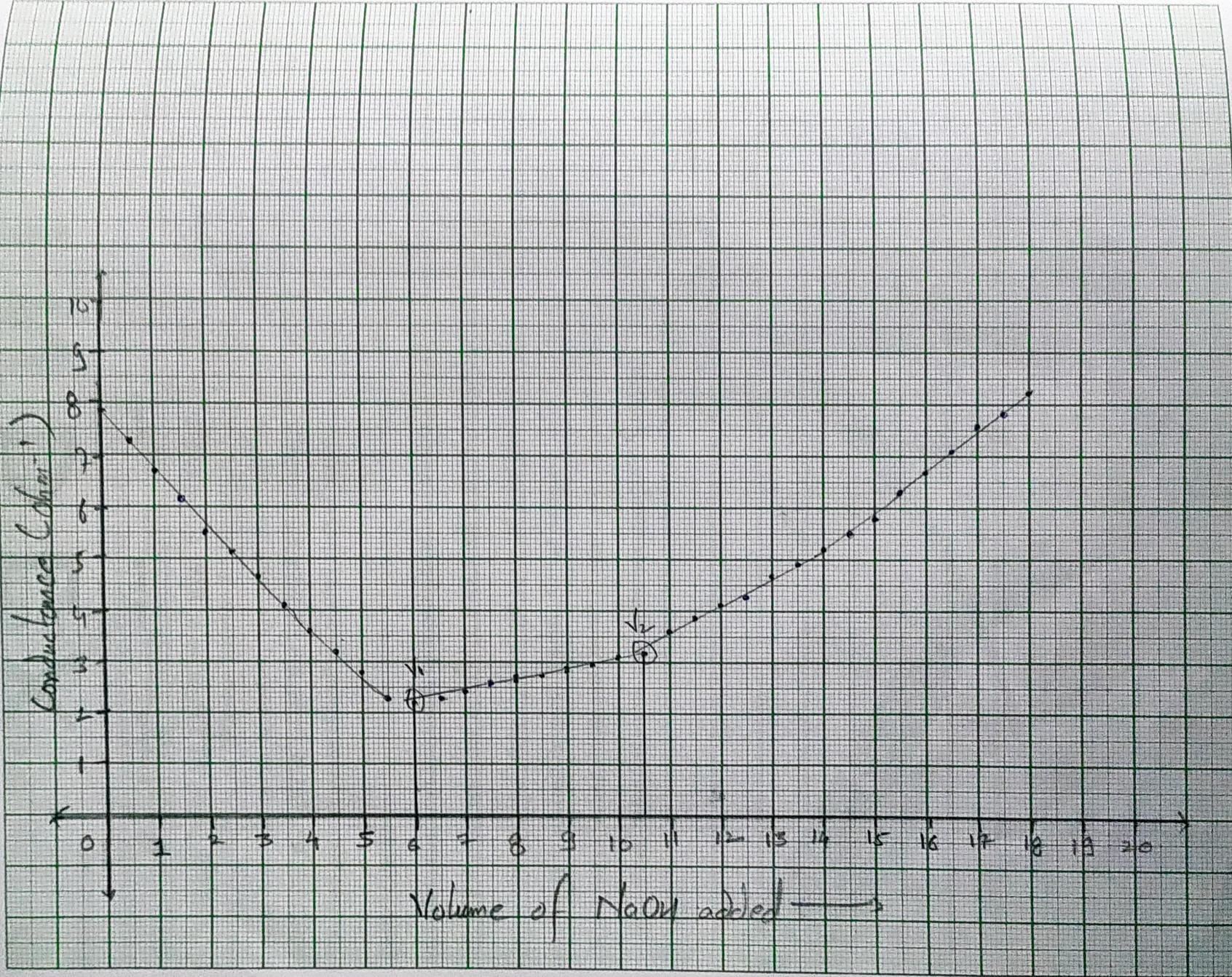
Strength of CH_3COONa

Volume of mixture = 20ml

Volume of NaOH = 11.5 - 6 = 5.5ml (From graph)

Normality of NaOH = 0.1

$$\text{Strength of } \text{CH}_3\text{COONa} = \frac{0.1 \times 5.5}{20} = 0.0275N$$



Experiment - 6

Aim

To estimate the amount of ferrous (Fe^{2+}) ions present in the given solution

Apparatus Required

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

Reagents Required

Ferrous ammonium Sulphate, dil H_2SO_4 , standard $\text{K}_2\text{Cr}_2\text{O}_7$

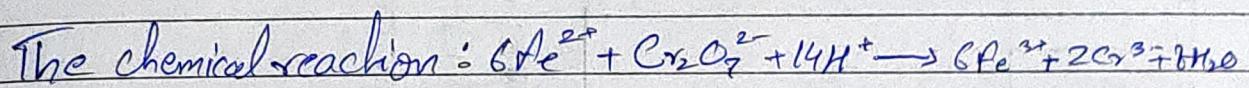
Principle

measurement of $E_{\text{m.p}}$ of an electrochemical cell using a potentiometer and change in $E_{\text{m.p}}$ due to the chemical (redox) reaction is monitored. In this potentiometer titration set up, an indicator electrode (Pt electrode) and reference (calomel) is coupled to form electrochemical cell for Fe^{2+} to Fe^{3+} . Fe^{2+} is oxidized to Fe^{3+} as $\text{K}_2\text{Cr}_2\text{O}_7$ is progressively added.

Platinum electrode which is kept in contact with a 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 varies to $[\text{Fe}^{2+}/\text{Fe}^{3+}]$ initially. During titration of Fe^{2+} in H_2SO_4 medium with $\text{K}_2\text{Cr}_2\text{O}_7$, this ratio varies to 0

little extent at the beginning and suddenly near the end point. It is observed that there is a sudden increase in the EMF of the cell at equivalence point.

The cell set up : $\text{Hg} | \text{HgCl}_2(\text{s}), \text{KCl}(\text{aq}) || \text{Fe}^{2+} | \text{Fe}^{3+}, \text{Pf}$



Result

The amount of ferrous ion present in the given solution is 0.734 g

Model graph

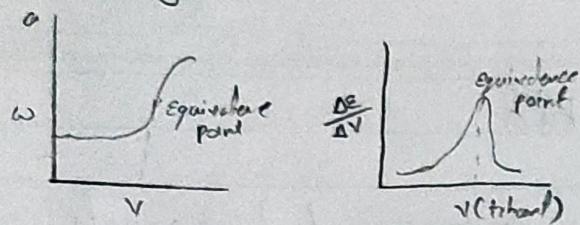


Table - I

S/No	Volume of $K_2Cr_2O_7$ solution (ml)	Emf (mV)
1	0	362
2	1	368
3	2	417
4	3	433
5	4	450
6	5	471
7	6	495
8	7	691
9	8	721
10	9	729
11	10	732
12	11	757
13	12	764
14	13	770
15	14	778

Table - 2

SL NO.	Volume of $K_2Cr_2O_7$ solution	E_mV/mV	$\Delta E(mV)$	$\Delta V(cm)$	$\Delta E/\Delta V$
1	4.8	448	—	—	—
2	5	456	8	0.2	40
3	5.2	460	4	0.2	20
4	5.4	464	4	0.2	20
5	5.6	468	4	0.2	20
6	5.8	472	3	0.2	15
7	6	477	5	0.2	25
8	6.2	480	3	0.2	15
9	6.4	488	8	0.2	40
10	6.6	493	5	0.2	25
11	6.8	501	8	0.2	40
12	7	514	13	0.2	65
13	7.2	525	11	0.2	85
14	7.4	564	39	0.2	195
15	7.6	608	104	0.2	520
16	7.8	685	10	0.2	50
17	8	699	9	0.2	45
18	8.2	708	7	0.2	35
19	8.4	715	6	0.2	30
20	8.6	721	3	0.2	15
21	8.8	726	4	0.2	20
22	8	728	6	0.2	30
23	9.2	734	6	0.2	30
24	9.4	740	6	0.2	30

Calculation

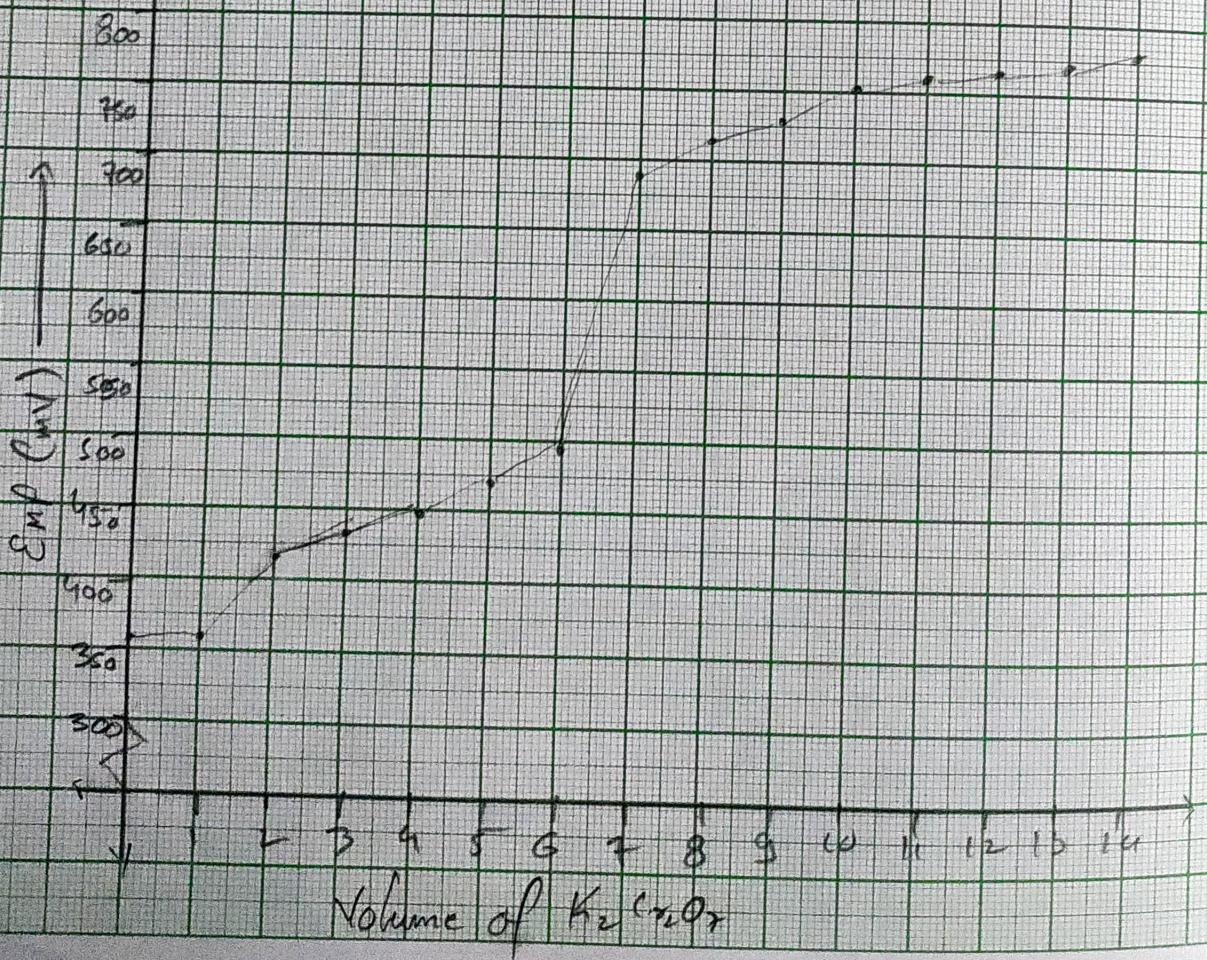
Volume of pipette solution = 10 ml

Volume of $K_2Cr_2O_7$ = 7.6 (From graph)

Normality of $K_2Cr_2O_7$ = 0.1 N

Strength of FAS, $\frac{0.1 \times 10}{7.6} = 0.1315\text{ N}$

Amount of Fe^{2+} in 100 ml = $\frac{55.85 \times 0.1315}{10} = 0.734\text{ g}/100\text{ ml}$



Size

1

150

180

170

160

150

140

130

120

110

100

90

80

70

60

50

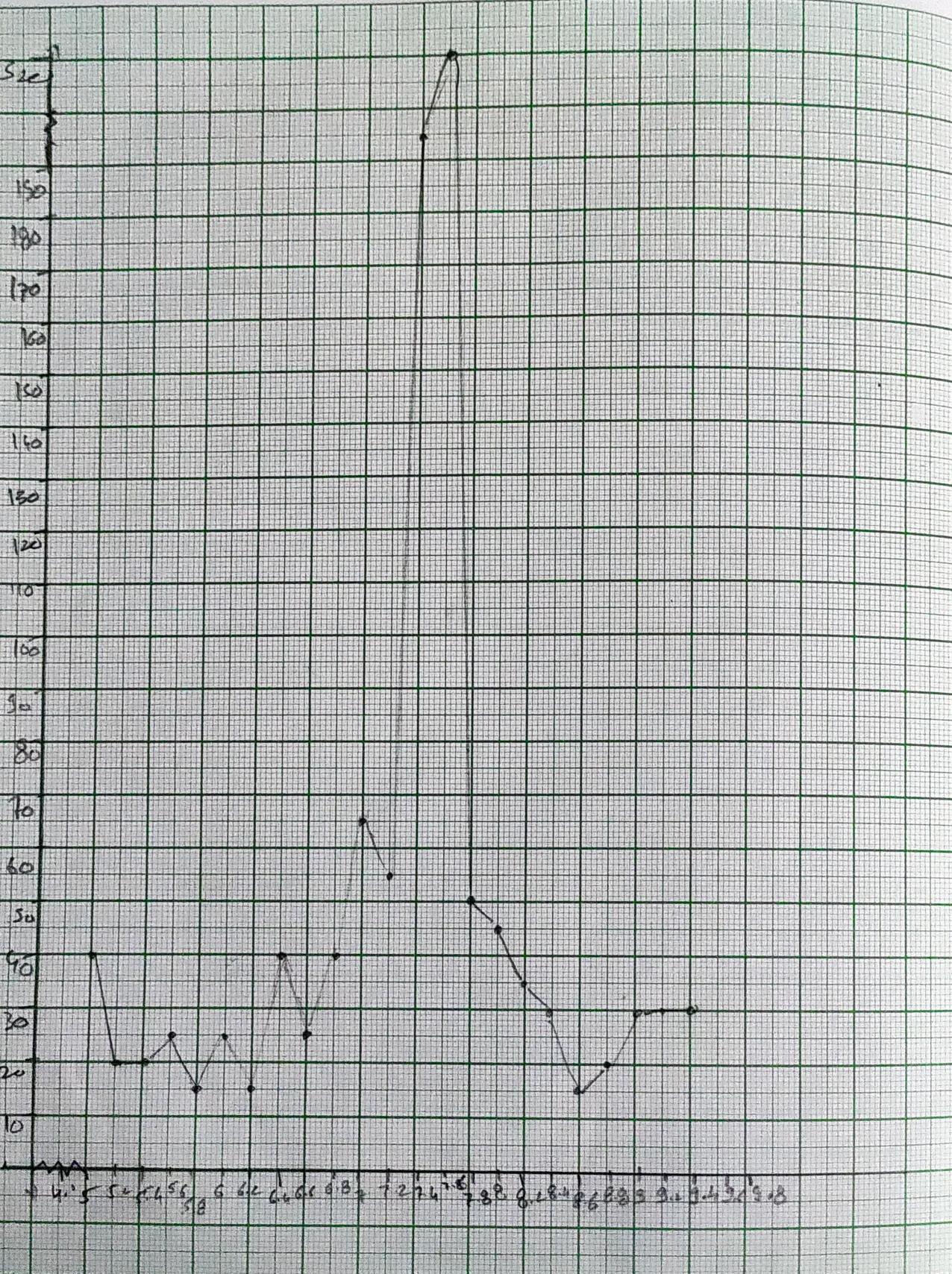
40

30

20

10

4.5 5.5 6.5 6.8 6.6 6.6 6.8 7.2 7.4 7.6 7.8 8.2 8.4 8.6 8.8 9.2 9.4 9.6 9.8



Experiment - 7

Aim

To find out the strength of given HCl by titrating it against NaOH (0.1N) using pH meter.

Apparatus Required

250 and 100 ml beaker, glass rod, 10 ml pipette, 100 ml standard measuring flask, pH meter

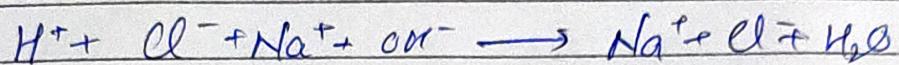
Reagents Required

NaOH solution (0.1N), HCl sample solution

Principle

A pH meter is a scientific instrument that measures the difference in electrical potential between a pH electrode and the reference electrode with respect to hydrogen ion concentration which is expressed as "pH value". Initially there is a little change in pH value due to the change in electrode potential that depends upon the fraction of H^+ ions removed by a base NaOH when added in small increments to the acid (HCl). As the end point is reached, the fraction of H^+ ions removed increases rapidly by the base that causes

change in p^H of the test solution



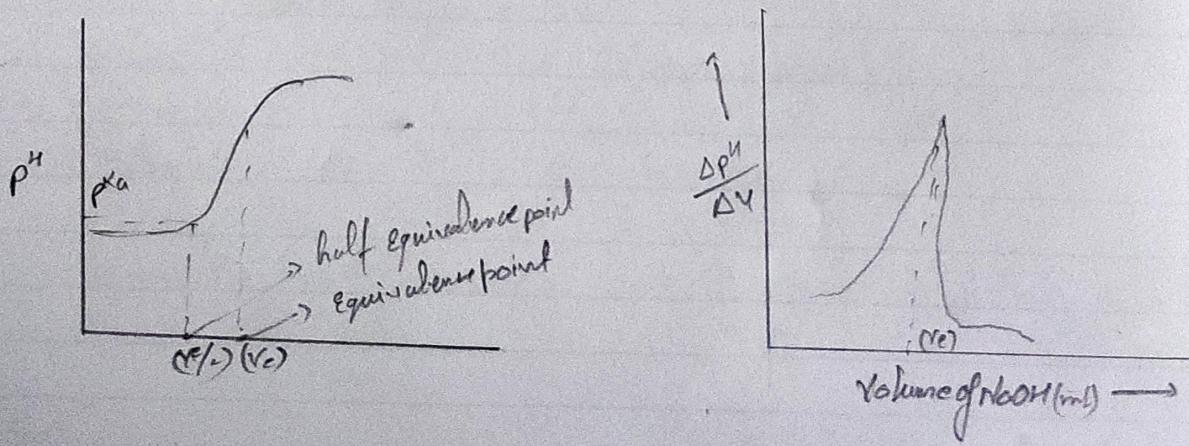
Result

The strength of the given HCl solution = 0.056 N

Table - 1

Sl No.	Volume of NaOH ml	pH
1	0	2.84
2	1	2.86
3	2	2.90
4	3	2.94
5	4	3
6	5	3.07
7	6	3.15
8	7	3.26
9	8	3.40
10	9	3.61
11	10	4.13
12	11	7.11
13	12	9.85
14	13	10.41
15	14	10.67
16	15	10.84

Modal graph



S.NO.	Volume of NaOH	pH	ΔpH	$\Delta V_{(ml)}$	$\Delta pH/\Delta V$
1	9	3.69	-	-	-
2	9.2	3.73	0.04	0.2	0.2
3	9.6	3.83	0.1	0.4	0.5
4	9.6	3.91	0.8	0.2	4
5	9.8	4.02	0.16	0.2	0.8
6	10	4.31	0.29	0.2	1.2
7	10.2	4.86	0.55	0.2	2.75
8	10.4	5.83	0.97	0.2	3.85
9	10.6	6.29	0.46	0.2	2.3
10	10.8	6.80	0.51	0.2	2.55
11	11	7.20	0.40	0.2	2
12	11.2	8.70	1.5	0.2	2.5
13	11.4	9.26	0.56	0.2	2.8
14	11.6	9.47	0.21	0.2	1.05
15	11.8	9.22	0.25	0.2	1.25
16	12	9.87	0.15	0.2	0.75
17	12.2	10.05	0.18	0.2	0.9
18	12.4	10.17	0.12	0.2	0.6
19	12.6	10.35	0.08	0.2	0.4
20	12.8	10.33	0.08	0.2	0.4
21	13	10.42	0.09	0.2	0.45
22	13.2	10.59	0.12	0.2	0.6
23	13.4	10.72	0.18	0.2	0.9

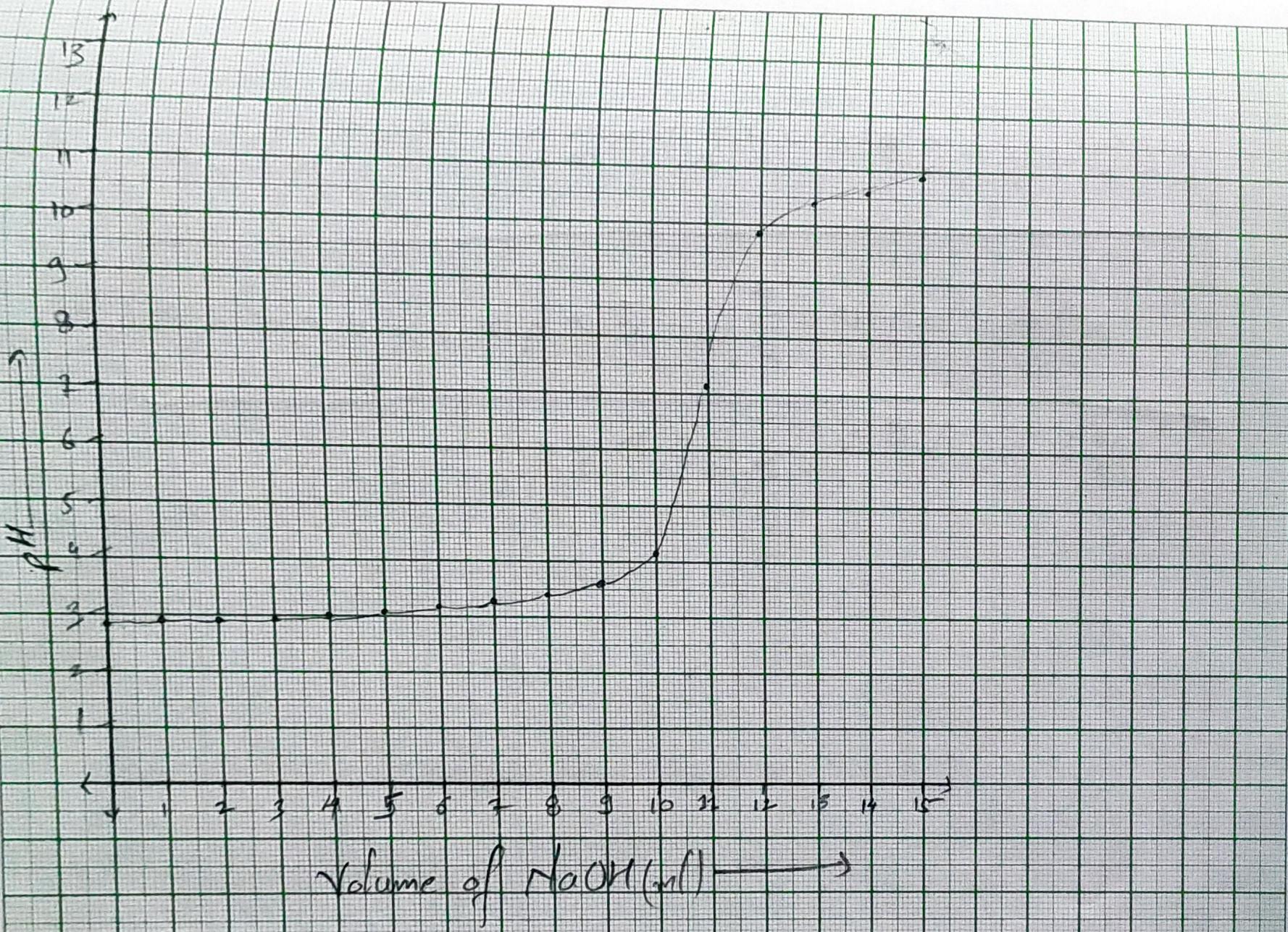
Calculation

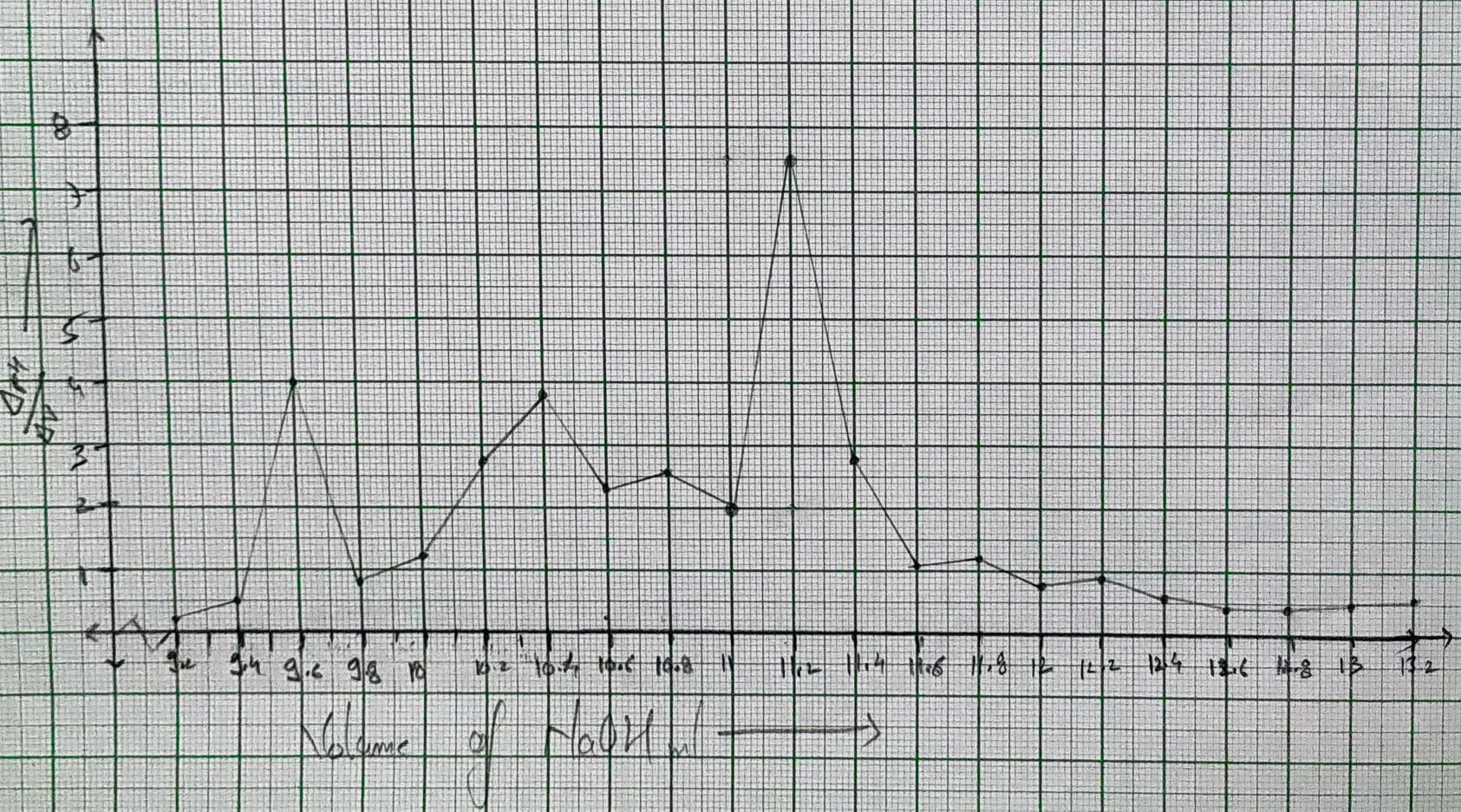
Volume of NaOH = 11.2 ml (From graph)

Strength of NaOH = 0.05N

Volume of HCl = 10 ml

$$\text{Strength of HCl} = \frac{11.2 \times 0.05}{10} = 0.056N$$





Experiment - 8

Aim

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

Apparatus required

Ostwald's viscometer, stopwatch, standard flask, graduated pipette and suction bulb

Reagents required

Polymer (PVA) and water

Principle

Measurement of solution viscosity offers a simple and convenient method for molecular weight determination if a polymer is soluble in a suitable solvent. In a capillary viscometer (Ostwald / Ubbelohde), 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. 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 pure solvent then

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

specific viscosity η_{sp} is defined as

$$\eta_{sp} = \frac{\eta - \eta_0}{\eta_0} = \frac{\eta}{\eta_0} - 1$$

$$= \eta_{red} - 1$$

Reduced viscosity η_{red} is equal to the ratio of the relative viscosity to concentration of the sample

$$\eta_{red} = \frac{\eta_{sp}}{c}$$

The Schulz-Mark-Houwink equation which relates η with molecular weight

$$\eta_i = K(m)^a$$

For polyvinyl alcohol solution, $K = 45.3 \times 10^{-3}$ and $a = 0.64$

where 'm' is molecular weight of the polymer, η_i is intrinsic viscosity, K is an empirical parameter characteristic of a particular solute-solvent pair and 'a' is a shape parameter. From known values of K and a , molecular weight can be determined

Intrinsic viscosity

A plot of η_{sp}/c vs c is a straight line for dilute solutions.

$$n_i = \lim_{c \rightarrow 0} \frac{m_p}{c}$$

Result

The molecular weight of the given polymer is 88.86 g

Modal graph

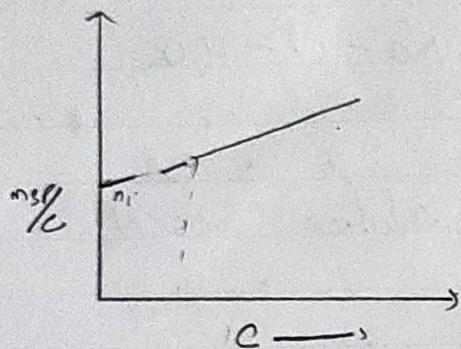


Table - 1

Sl No.	Conc. of Polymer Soln	Time of flow (in sec)	Relative viscosity	specific viscosity	Reduced viscosity
1	Pure solvent	38 (t ₀)	—	—	—
2	0.1	43	1.131	0.131	1.31
3	0.2	46	1.210	0.210	1.05
4	0.3	50	1.315	0.315	1.06
5	0.4	55	1.447	0.447	1.118
6	0.5	62	1.631	0.631	1.263

Calculation

$$n_{int} = K \times m^\alpha$$

$$\log n_{int} = \log K + \alpha \log m$$

$$\alpha \log m = \log n_{int} - \log K$$

$$\log m = \frac{\log n_{int} - \log K}{\alpha}$$

$$m = \text{Anti log} \left[\frac{\log n_{int} - \log K}{\alpha} \right]$$

$$n_{int} = 0.8$$

$$K = 45.3 \times 10^{-3}$$

$$\alpha = 0.64$$

$$m = 88.86233 \text{ g}$$

