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EXPERIMENT NO. 1 (Estimation of total, permanent & temporary hardness by EDTA method)

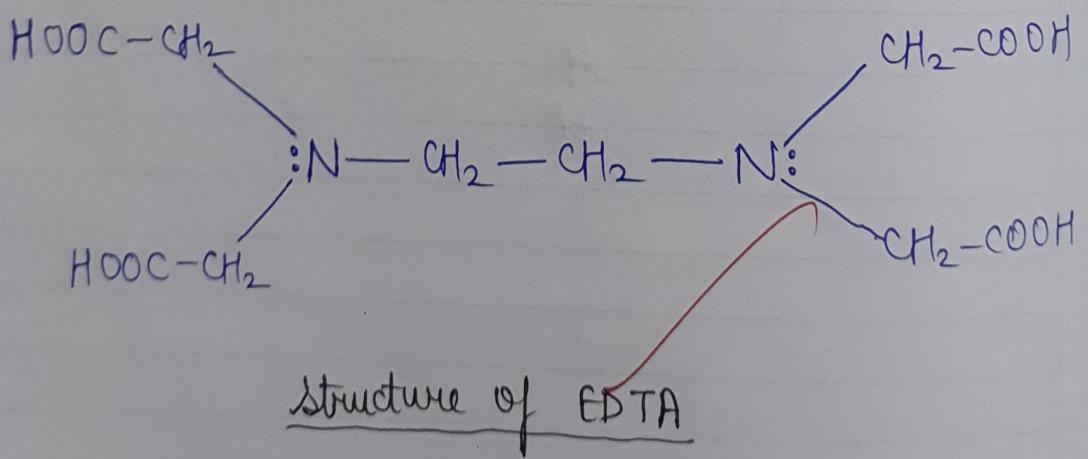
Aim: To estimate the amount of total hardness, permanent hardness and temporary hardness of a given sample of water by EDTA method using ammonia buffer ($\text{pH} = 10$) as EBT (Eriochrome Black T) indicator.

Apparatus Regd: Burette, pipette, conical flask, standard volumetric flask, funnel, beaker, beaker (250 ml).

Reagents Regd: EDTA solution, standard hard water, sample water, Eriochrome Black-T indicator (EBT), $\text{NH}_3 - \text{NH}_4\text{Cl}$ buffer solution ($\text{pH} = 9-10$).

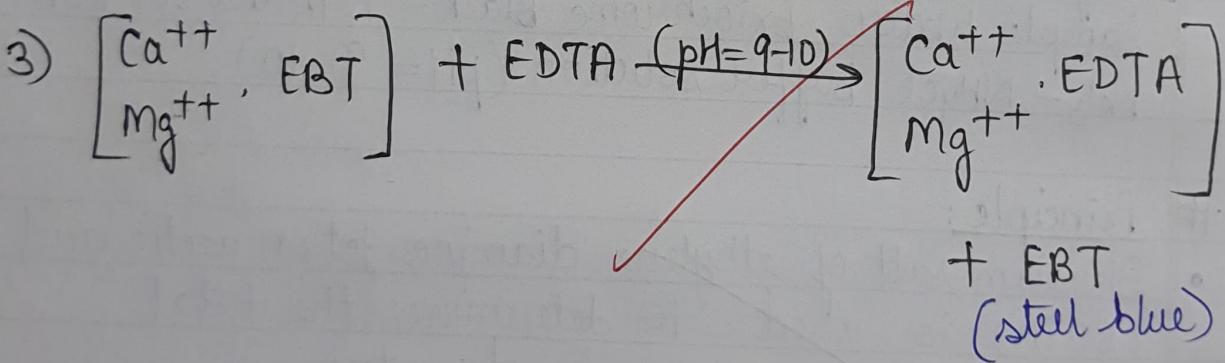
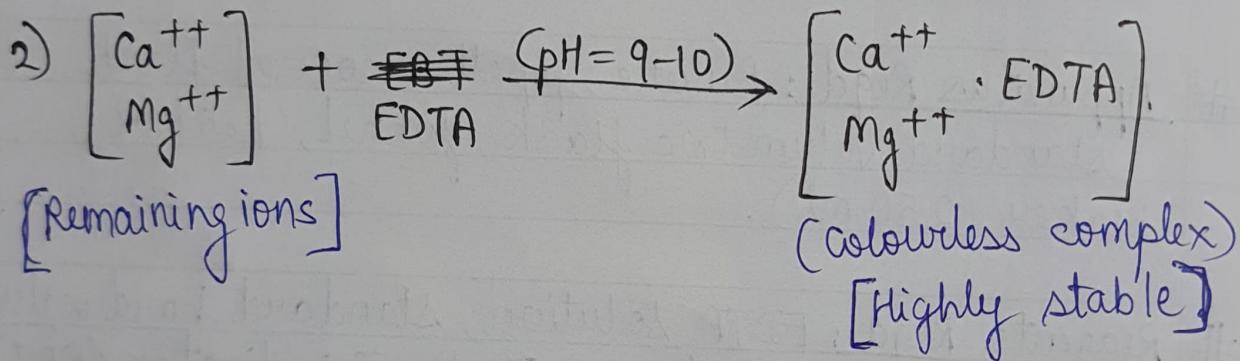
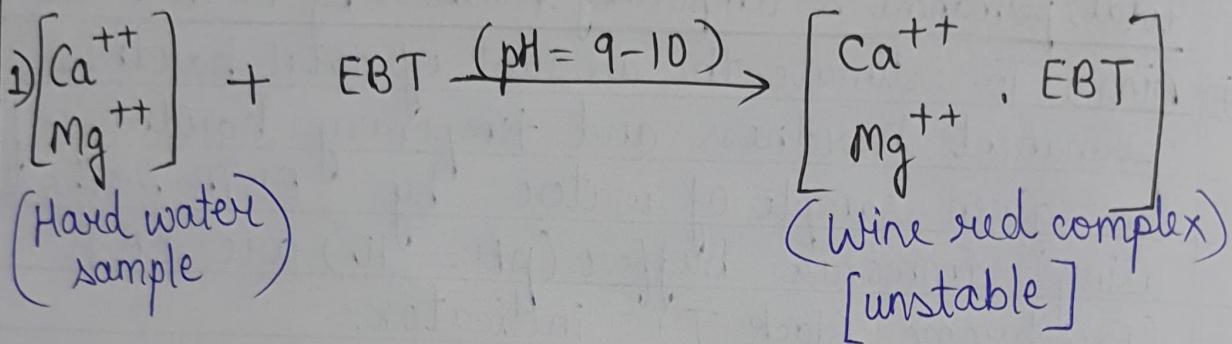
Principle:

- 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 EBT (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 EBT indicator, wine red colour changes to steel blue. This is the end point for the titration b/w EDTA and hard water.
- The metal indicator complex is less stable than metal EDTA complex. So this metal indicator complex is titrated with std. EDTA solⁿ. Now, colourless metal - EDTA complex is formed by releasing EBT indicator which is blue in colour. So the colour change from wine red to blue indicates the end point.
- When the sample water is boiled, bicarbonates of calcium & 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 EBT indicator.

► Reactions



Procedure★ Preparation of standard hard water

Dissolve 1gm of CaCO_3 (dry) in minimum (2-3 drops) quantity of dil. HCl and evaporate the solⁿ on water bath. Dissolve this solⁿ in small amount of distilled water and then transfer into 1000 ml std. flask. Make up the solⁿ upto the mark and shake well for uniform concentration.

★ Standardization of EDTA

~~Pipette out 20 ml of standard hard water into a clean conical flask. Add 5 ml of the buffer solⁿ and 3 or 4 drops of the EBT indicator. The solⁿ turns wine red in colour. Titrate the wine red coloured solⁿ 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 V_1 ml be the volume of EDTA consumed.~~

Observations:

1) Standardization of EDTA (std. Hard water v/s EDTA soln)

SNO	Vol of std. hard water	Burette reading Initial	Burette reading Final	Vol of EDTA used.	Indicator
1	20 ml	0	20.3	20.3	
2	20 ml	20.3	40.3	20	EBT
3	20 ml	40.3	60.3	60.3 20	

(Concordant Volume = 20 ml)

★ # Calculations :

1 ml of std. hard water = 1 mg of CaCO_3

Volume of std. hard water taken = 20 ml

20 ml of std. hard water = 20 mg of CaCO_3

Volume of EDTA consumed (V_1) = 20 ml

V_1 ml EDTA solution [i.e. 20 ml] = 20 mg CaCO_3

\therefore 1 ml EDTA solution = $20/V_1$ mg of equivalent CaCO_3

* Determination of Total Hardness

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

(P.T.O) →

Observations:

2) Determination of Total Hardness (Sample Hard Water v/s EDTA soln)

S.NO	Vol. of sample hard water	Burette Reading Initial	Burette Reading Final	Vol. of EDTA reqd.	Indicator
1	20ml	0	4.7	4.7	
2	20ml	4.7	9.2	4.5	EBT
3	20ml	9.2	13.7	4.5	

(Concordant Volume = 4.5 ml)

Calculations:

Volume of EDTA consumed (V_2) = 4.5 ml

Now, if 1 ml EDTA = $20/V_1$ mg CaCO_3

Then, V_2 ml EDTA = $\frac{20}{V_1} \times V_2$ mg CaCO_3

If 20 ml sample hard water taken for titration contains = $\frac{20}{V_1} \times V_2$ mg CaCO_3

Then, 1000 ml (i.e. 1L) will contain = $\frac{20}{V_1} \times V_2 \times \frac{1000}{20}$ mg CaCO_3
 $= \frac{V_2}{V_1} \times 1000$ ppm

Total hardness = $\left(\frac{4.5}{20} \times 1000 \right)$ ppm
 $= 225$ ppm

* Determination of Permanent Hardness

Take 100 ml 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 V_2 ml be the volume of EDTA consumed.

* Estimation of temporary hardness

$$\begin{aligned}\text{Temporary hardness} &= \text{Total hardness} - \text{permanent hardness} \\ &= (225 - 60) \text{ ppm} \\ &= 165 \text{ ppm}\end{aligned}$$

Result:

- Total hardness of sample hard water = 225 ppm
- Permanent hardness of sample hard water = 60 ppm
- Temporary hardness of sample hard water = 165 ppm

Observations :

3) Determination of permanent hardness (Boiled water)
 [sample v/s EDTA soln]

S.NO	vol. of std. hard water	Burette Reading Initial	Reading Final	vol. of EDTA reqd.	Indicator
1	20ml	0	1.4	1.4	
2	20 ml	1.4	2.6	1.2	EBT
3	20ml	2.6	3.8	1.2	

(Concordant Volume = 1.2 ml)

Calculations :

Volume of EDTA consumed (V_3) = 1.2 ml

Now, if 1 ml EDTA = $20/V_1$ mg CaCO_3

Then, V_3 ml EDTA = $\frac{20}{V_1} \times V_3$ mg CaCO_3

If 20 ml sample hard water taken for titration

contains = $\frac{20}{V_1} \times V_3$ mg CaCO_3

Then, 1000 ml (i.e. 1L) will contain = $\frac{20}{V_1} \times V_3 \times \frac{1000}{20}$ mg CaCO_3
 $= \frac{V_3}{V_1} \times 1000$ ppm

Permanent hardness = $\left(\frac{1.2}{20} \times 1000 \right)$ ppm
 $= 60$ ppm

EXPERIMENT NO-2

(Determination of the amount of Na_2CO_3 and NaOH in a given mixture using hydrochloric acid)

* AIM: To determine the amount of Na_2CO_3 and NaOH in a given mixture using hydrochloric acid.

* Apparatus Regd: Burette, pipette, conical flask, std. volumetric flask, funnel, beaker 250ml, measuring cylinder.

* Reagents Regd : N/50 Na_2CO_3 , Methyl Orange, phenolphthalein, water sample, HCl.

* 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.

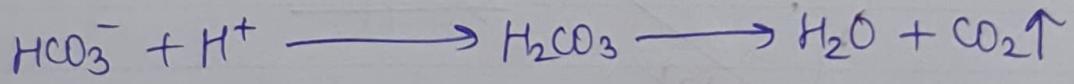
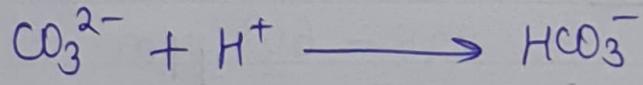
$$A = \text{all hydroxide ions} + \text{half of carbonate ions}$$

$$B = \text{half the carbonate ions after phenolphthalein end pt.}$$

$$2B = \text{all carbonate ions}$$

$$A - B = \text{all hydroxide ions.}$$

★ Reactions :



★ Procedure

➤ Titration-I : Standardization of HCl

20 ml of 0.02 N Na_2CO_3 solⁿ is pipetted into a clean conical flask and 2 or 3 drops of methyl orange indicator is added to the solⁿ. The solⁿ is then titrated against HCl taken in the burette. The end pt. is change of colour from yellow to orange. The titrations are repeated to get concordant value.

★ Observations

Table-1: Standardization of HCl

S.NO	Volume of Na ₂ CO ₃ soln (in ml)	Burette reading:		Concordant Volume of HCl reqd.	Indicator
		Initial	final		
1	20 ml	0	18.1	18.1	
2	20 ml	18.1	36.1	18	Methyl orange
3	20 ml	36.1	54.1	18	

(Concordant volume = 18 ml)

★ Calculations

$$\text{Volume of HCl} (V_1) = \underline{18} \text{ ml (end point)}$$

$$\text{Let normality of HCl} = N_1$$

$$\text{Vol. of Na}_2\text{CO}_3 (V_2) = \underline{20} \text{ ml}$$

$$\text{Normality of Na}_2\text{CO}_3 (N_2) = \underline{0.02} N$$

$$\therefore N_1 V_1 = N_2 V_2$$

$$\Rightarrow \text{Normality of HCl} (N_1) = \frac{N_2 V_2}{V_1} = \frac{(0.02)(20)}{(18)}$$

$$= \frac{1}{45} N = \underline{0.022} N$$

► Titration-II: Titration of Na_2CO_3 and NaOH in a given mixture

The given unknown solⁿ is made upto 100ml in a standard flask using distilled water. 20ml of this made up solⁿ is pipetted into a clean conical flask. Add 2 to 3 drops of phenolphthalein indicator & titrate it against std. HCl. The disappearance of pink colour is observed as end point and note down the titre value. Let it be A ml. To the same solⁿ, add 2 to 3 drops of methyl orange indicator and continue the titration till the colour 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.

$$\therefore N_1 V_1 = N_2 V_2$$

$$\text{Normality of mixture (N}_2) = \frac{N_1 V_1}{V_2} = \frac{2B \times N_L}{20} = \frac{14 \times 0.022}{20} = 0.015 \text{ N}$$

$$\text{Amount of } \text{Na}_2\text{CO}_3 \text{ present in whole of the given sol}^n = N_2 \times \frac{53}{10}$$

To calculate ppm
 $N \times E \times 1000$

$$= 0.015 \times \frac{53}{10}$$

$$= 0.08 \text{ g}/100\text{ml}$$

Teacher's Signature:

$$= (0.015 \times \frac{53}{10} \times 1000) \text{ ppm}$$

$$= 795 \text{ ppm}$$

★ Observations

Table- 2: Estimation of Na_2CO_3 and NaOH in a given mixture

S.NO	Volume of the unknown soln (in ml)	Initial Burette Reading (ml)	Volume consumed for phenolphthalein end point (A) Burette Reading (ml)	Volume consumed for methyl orange end point after ph end point. (B) Burette Reading (ml)
1	20	0	11	7.5
2	20	0	10.5	7
3	20	0	10.5	7

(Concordant value = 10.5) (Concordant value = 7)

★ Calculations

I: Estimation of the amount of Na_2CO_3

$$\text{Vol. of HCl (V}_1\text{)} = 2B \quad (\text{B is the titre value after phenolphthalein end point})$$

$$= 2 \times 7 \\ = 14 \text{ ml}$$

$$\text{Normality of HCl (N}_1\text{)} = 0.022 \text{ N}$$

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

★ Result:

The amount of Na_2CO_3 present in the given solⁿ

$$= 0.08 \text{ g / 100ml}$$

$$= 795 \text{ ppm}$$

The amount of NaOH present in the given solⁿ

$$= 0.016 \text{ g / 100ml}$$

$$= 160 \text{ ppm}$$

II: Estimation of amount of NaOH

$$\text{Vol. of HCl (V}_1\text{)} = (\text{A}-\text{B}) \text{ ml} = 3.5 \text{ ml}$$

$$\text{Normality of HCl (N}_1\text{)} = 0.022 \text{ N}$$

$$\text{Vol. of mix. (V}_2\text{)} = 20 \text{ ml}$$

$$\text{Normality of mixture (N}_2\text{)} = \frac{(\text{A}-\text{B}) \times \text{N}_1}{\text{V}_2}$$

$$= \frac{3.5 \times 0.022}{20}$$

$$= 0.004 \text{ N}$$

Amount of NaOH present in whole of the given solⁿ

$$= \text{N}_2 \times \frac{40}{10}$$

$$= 0.004 \times \frac{40}{10}$$

$$= \underline{\underline{0.016 \text{ g / 100ml}}}$$

OR $\text{N}_2 \times 1000 \text{ ppm}$

$$= (0.004 \times 40 \times 1000) \text{ ppm}$$

$$= \underline{\underline{160 \text{ ppm}}}$$

EXPERIMENT NO - 3

(Determination of molecular weight of a polymer by viscosity average method)

* Aim: To determine the molecular weight of a polymer by using Ostwald's viscometer.

* Apparatus Required: Ostwald's viscometer, burette, conical flask, standard volumetric flask, funnel, beakers, graduated pipettes, stopwatch; etc.

* Reagents Required: Distilled water, polymer solⁿ of different concentrations.

* Principle:

If a polymer is sol^b in a suitable solvent, measurement of solⁿ viscosity provides a simple & 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. Molecular weight of polymers can be determined by viscosity measurements.

1. Preparation of binary system for two in all
of 80% -
2. moving off of the ring 11001 for toluene and
benzene ratio 0 -

Polymer	Solvent	K value	α value
cellulose acetate	Acetone	1.5×10^{-4}	0.82
Poly Vinyl Alcohol	Water	45.3×10^{-3}	0.64
polystyrene	Toluene	3.7×10^{-4}	0.62
Polymethyl methacrylate	Benzene	0.95×10^{-4}	0.76

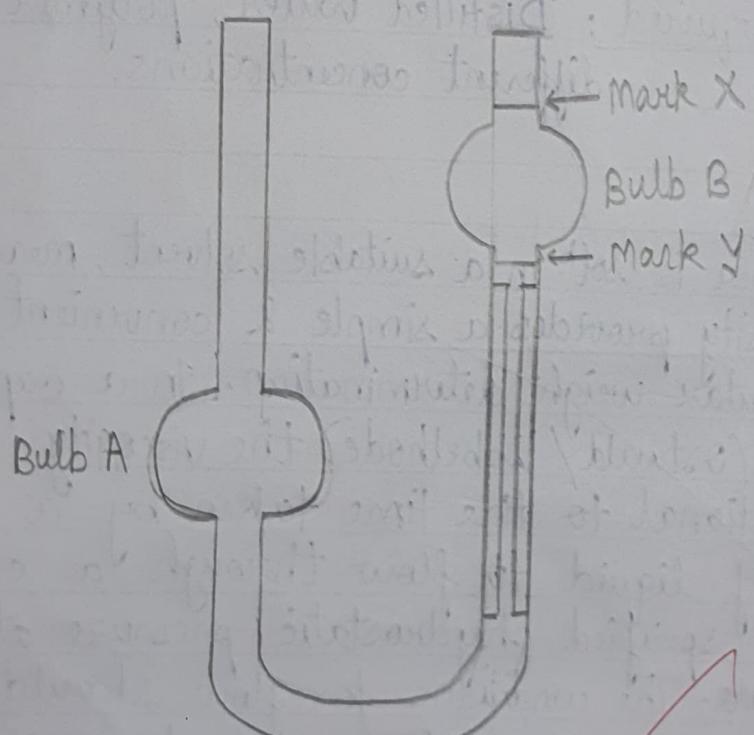
using Ostwald's viscometer, relative viscosity can be found out as

$$\eta_r = \eta / \eta_0$$

where η is the viscosity of polymer solⁿ and η_0 is the viscosity of pure solvent in which the polymer solⁿ is prepared. In this method, the liquid is allowed to flow through the capillary tube and the time taken for flowing from upper to lower mark in the viscometer is noted. This time is used to calculate the viscosity and the molecular weight of the polymer. Acc-to Mark and Houwink relation b/w intrinsic viscosity, $[\eta]$ & molecular weight, M,

$$[\eta_{int}] = KM^\alpha$$

If intrinsic viscosity of a polymer is known, then it's molecular weight can be calculated using above eqn in which K and α are constants for a specific polymer and a specific solvent. " α " is a scalar which relates to the "stiffness" of the polymer chains. If in solⁿ, the polymer molecules are rigid rods, then $\alpha=2$. On the other extreme, if the polymers are hard sphere, $\alpha=0$. If $\alpha=1$, the polymers are semi-coils. In a Flory Theta solvent, $\alpha=0.5$, and in a



thermodynamically good solvent, $\alpha = 0.8$. To determine $[\eta]$, viscosities of several dilute solutions of a polymer in a solvent as well as η_0 are measured & the values are plotted as either reduced viscosity, η_{red} or inherent viscosity, η_{inh} . i.e. $\ln(\eta/\eta_0)$ versus concentration, c . Extrapolation to zero polymer concentration eliminates polymer intermolecular interactions. The curves of both plots should be linear and have a common intercept that is the intrinsic viscosity.

★ Procedure:

[Part - 1] : To determine the time of flow for pure solvent.

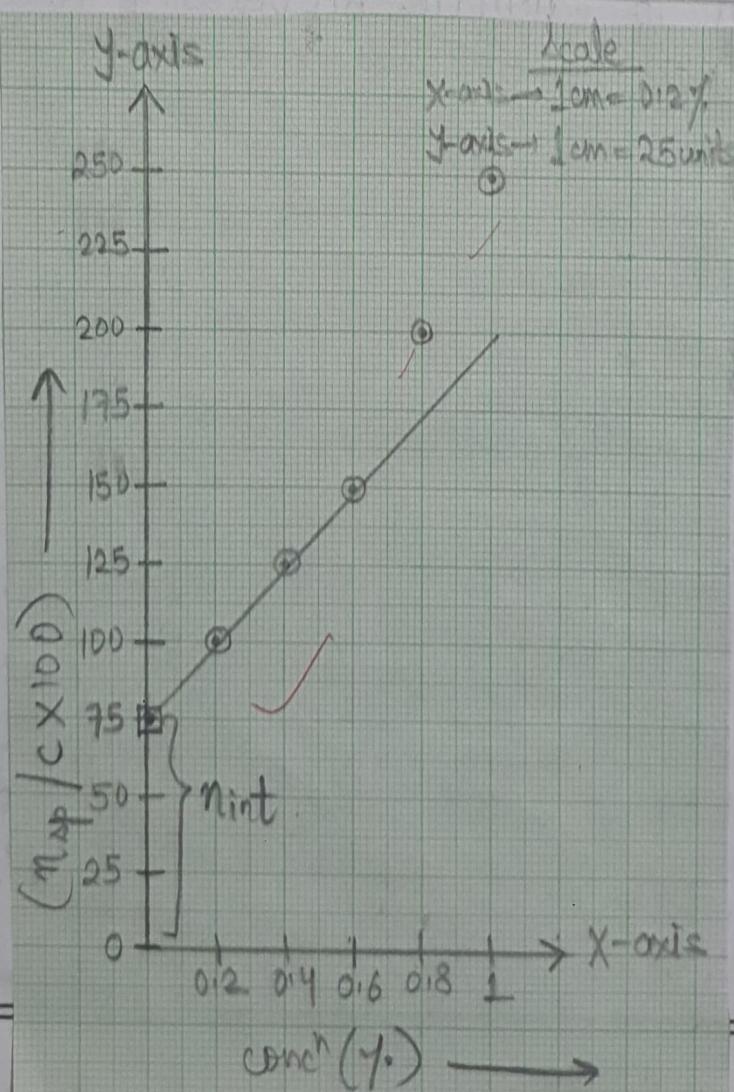
Take a clean & dry Ostwald viscometer and clamp the wide arm of the viscometer to the retort stand such that it should be perfectly vertical in position. Introduce 15 ml of pure solvent into the wide arm such that the liquid level should not exceed the mark "G" and then attach a rubber tube to the narrow arm of the viscometer. Now suck the pure solvent by the rubber tube till the solvent level rises above the upper mark "E" of narrow arm above the bulb. Allow the solvent to flow down the capillary tube & note the time (using stop watch) in seconds taken

★ Observations

Table-1: determination of molecular weight of polymer

Solvent/polymer solution	Time t in sec for polymer soln & t_0 for pure solvent (avg).	$\eta_{sp} = \eta / \eta_0$ or $\eta_{sp} = t / t_0$	$\eta_{sp} = \eta_{sp} - 1$	$n_{red} = \eta_{sp}/c$	$\eta_{sp}/c \times 100$
Pure solvent	$t_0 = 92.0$	$92/92 = 1$	$1 - 1 = 0$	0	0
0.2% polymer soln	$t_1 = 111.5$	$111.5/92 = 1.2$	$1.2 - 1 = 0.2$	1	100
0.4% polymer soln	$t_2 = 145.0$	$145/92 = 1.5$	$1.5 - 1 = 0.5$	1.25	125
0.6% polymer soln	$t_3 = 178.5$	$178.5/92 = 1.9$	$1.9 - 1 = 0.9$	1.5	150
0.8% polymer soln	$t_4 = 248.0$	$248/92 = 2.6$	$2.6 - 1 = 1.6$	2.0	200
1% polymer soln	$t_5 = 322.0$	$322/92 = 3.5$	$3.5 - 1 = 2.5$	2.5	250

★ graph



by the solvent to flow from upper mark to lower mark "F" on the narrow arm below the bulb of the viscometer. Repeat the procedure for two more times & take out the mean of the 3 readings i.e., meantime in seconds for pure solvent.

(Part-2) → To determine the time of flow for a given polymer (Poly Vinyl Alcohol) solⁿ.

Introduce 15 ml of 0.1% polymer solⁿ in a dry viscometer and repeat the procedure as described in part (1), to find out the mean time of flow in seconds. Similarly, repeat the procedure for 0.2, 0.4, 0.6 and 0.8% polymer solⁿ's & note the time of flow for each of the solⁿ 3 times. Then find the mean time in seconds for each polymer solⁿ. Before measuring the time of flow for each concⁿ (polymer solⁿ), the viscometer should be cleaned with distilled water & dried with acetone.

★ Result:

The molecular weight of given polymer poly vinyl alcohol is 105438.7.

Teacher's Signature:.....

★ Calculations:

Show calculations of η_r , η_{sp} , η_{red} and η_{int} for all solⁿ.

Extrapolate the straight line to Y-axis.

The intercept is $\eta_{int} = 75$ (from graph)

Using appropriate values of K, α and $[\eta]$ value obtained from graph; calculate the molecular weight of the given polymer.

$$[\eta_{int}] = KM^\alpha$$

$$M^\alpha = [\eta_{int}] / K$$

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

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

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

$$M = \text{antilog} [(\log \eta_{int} - \log K) / \alpha]$$

$$M = \text{antilog} [\{\log 75 - \log (45.3 \times 10^{-3})\} / 0.64]$$

$$M = \text{antilog} [\{1.875 - 1.656 + 3\} / 0.64]$$

$$M = \text{antilog} [3.219 / 0.64]$$

$$M = \text{antilog} (5.023)$$

$$M = 105438.7$$

EXPERIMENT NO-4

(Conductometric Titrations - Determination of strength of HCl (Strong Acid v/s Strong Base))

- ★ Aim: To determine the strength of a given solution of HCl by conductometric titration with a given NaOH solution.
- ★ Apparatus Regd: conductivity meter, conductivity cell, glass rod, beakers, burette, pipette, standard flask.
- ★ Reagents Regd: 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

Table-1: Standard NaOH v/s Unknown HCl (Titration)

S.NO	Vol. of NaOH (ml) added	Conductance in ohm ⁻¹
1	0	7.0
2	1	5.8
3	2	4.5
4	3	3.8
5	4	2.9
6	5	2.2
7	6	2.3
8	7	2.5
9	8	2.6
10	9	2.8
11	10	3.0
12	11	3.4
13	12	3.8

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 100ml in a standard flask. Pipette out 10ml of the made up HCl in to a beaker. Dilute the solⁿ with distilled water, so that the conductivity cell can be immersed well in the solⁿ (100ml). 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.

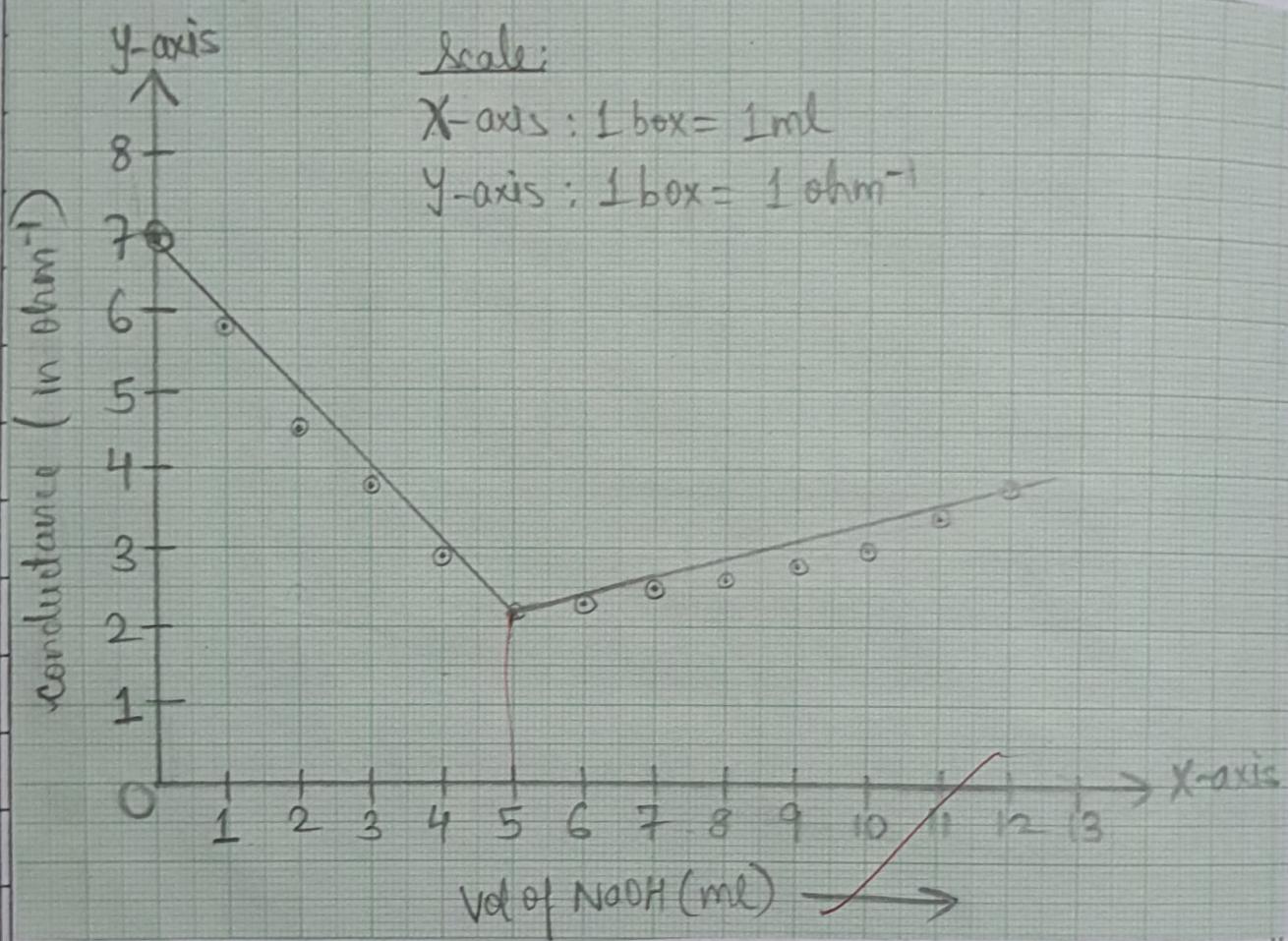
After each addition, stir the contents of the beaker and after an equilibrium time of 2-3 minutes, note the corresponding

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 100ml in a standard flask. Pipette out 10ml of the made up HCl in to a beaker. Dilute the solⁿ with distilled water, so that the conductivity cell can be immersed well in the solⁿ (100ml). 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 2 ml) with gentle stirring of the contents of the beaker.

After each addition, stir the contents of the beaker and after an equilibrium time of 2-3 minutes, note the corresponding



▲ graph based on Pilot Titration

conductance value and tabulate it. Continue the titration till atleast 10 increments, after the conductance reaches a minimum & starts increasing. After the completion of titration, wash the conductance cell with distilled water and immersed in water. Plot a graph b/w conductivity against volume of NaOH added. The intersection of two lines to the volume axis gives the end point. Volume of NaOH reqd. for neutralisation is taken from graph (fair) titration intersection pt. 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.

~~# Results~~

~~The strength of the given HCl solution = 200.25 g/dl~~

P.T.O. →

Table-2: Standard NaOH v/s Unknown HCl (Farr titration)

S.NO	Volume of NaOH(ml) added	conductance in ohm ⁻¹
1	0.0	7.0
2	1.0	6.0
3	2.0	4.9
4	3.0	4.2
5	4.0	3.3
6	4.2	3.1
7	4.4	2.9
8	4.6	2.8
9	4.8	2.6
10	5.0	2.4
11	5.2	2.2
12	5.4	2.1
13	5.6	2.1
14	5.8	2.5
15	6.0	3.2
16	7.0	3.8
17	8.0	4.3
18	9.0	4.9

Calculations :-

Volume of NaOH (V_2) = 5.5 ml (obtained from graph)

Normality of NaOH (N_2) = 0.1 N

Volume of HCl (V_1) = 10 ml

$$\text{Normality of HCl} (N_1) = \frac{N_2 V_2}{V_1} = \frac{0.1 \times 5.5}{10} = 0.055 \text{ N}$$

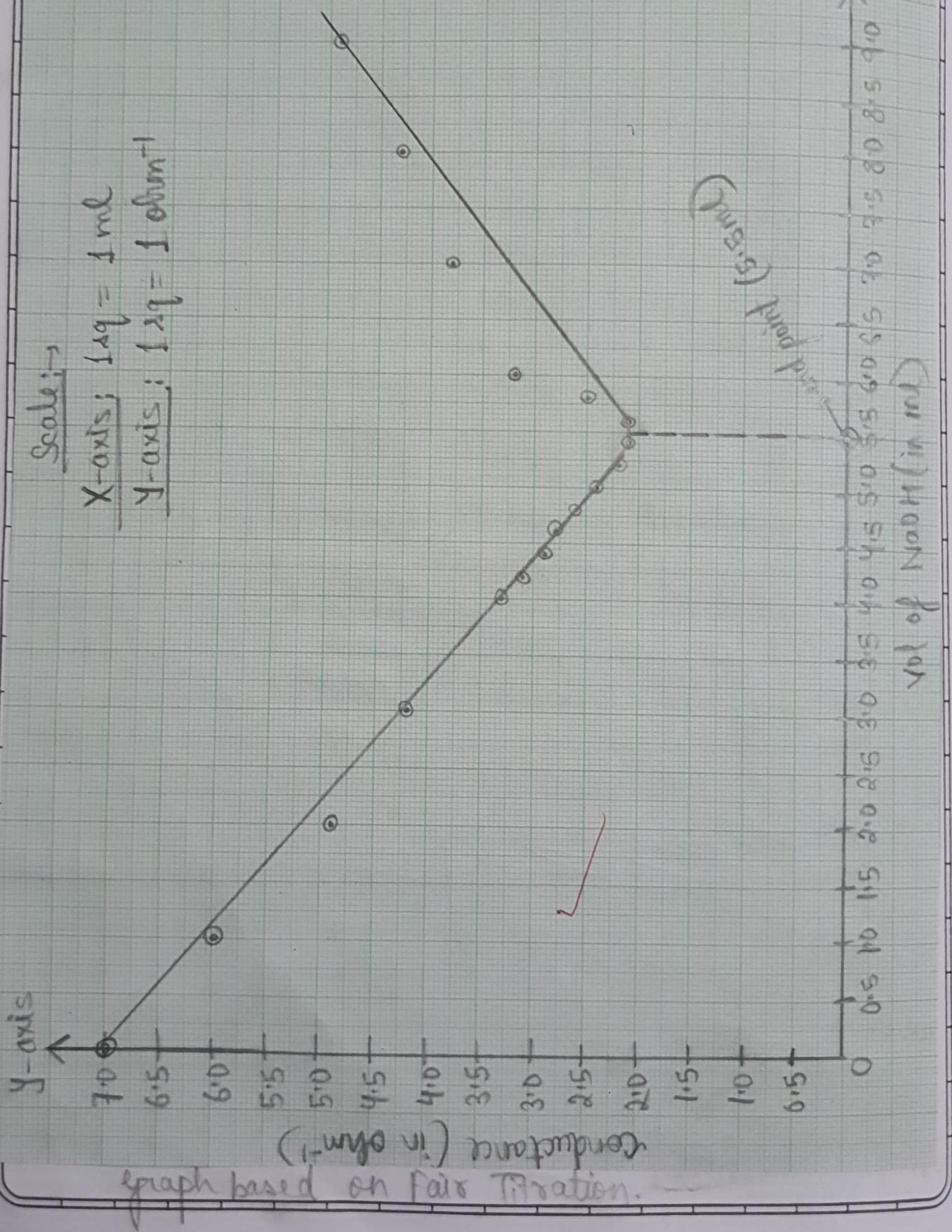
~~Strength~~ Strength of HCl = $(0.055 \times 36.5) \text{ g/L} = 2.0075 \text{ g/L}$

#

Result :→

The strength of the given HCl solution = 2.0075 g/L

obj/122



Experiment No 5

(determination of the strength of a mixture of acetic acid & HCl by conductometry)

★ Aim :→ To determine the strength of a mixture of acetic acid and HCl by conductometry.

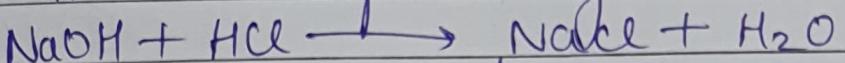
★ Apparatus Req'd :→ Conductivity meter, conductivity cell, glass rod, beakers, burette, pipette, standard flask.

★ Reagents Req'd :→ NaOH, HCl and CH_3COOH .

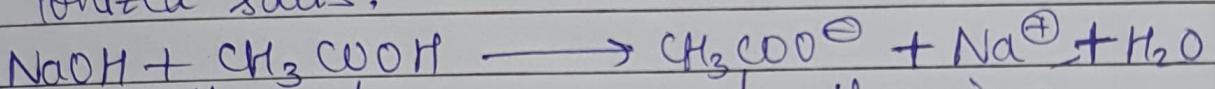
★ Principle :→ The principle of conductometric titrations is based on the fact that during the titration, one of the ions is replaced by the other ions and invariably these two ions differ in the ionic conductivity therefore, the conductivity of the solution varies during the course of titration.

- The equivalence point may be located graphically by plotting the change in conductance as a function of volume of titrant added.
- When a mixture of acids like a strong acid (HCl) and weak acid (CH_3COOH) are titrated against a strong base (NaOH), strong acid reacts first followed by a weak acid.

- When the titration of the strong base v/s a strong acid is carried out, there is a decrease in the conductivity as highly mobilized hydrogen ions are replaced by solution ions.



- When the whole strong acid is consumed, base reacts with weak acid & conductivity increases as unionized weak acid becomes the ionized salts.



- After both acids are consumed, there is a step increase in conductivity which gives the end point and the increase in conductivity is due to the fast-moving hydroxyl ions from the base added.

Procedure :-

The given mixture of acids is made upto 100 ml using distilled water. 10 ml of this made up solⁿ is pipetted out into clean beaker and 100ml of distilled water is added. The conductivity is ~~added~~ dipped into the test solⁿ and titrated against NaOH (0.5ml) interval with propn stirring. The conductance is measured after each 0.5 ml addition of NaOH at various stages of

★ Observation Table

S.NO	Volume of NaOH added (ml)	conductance (ohm ⁻¹)
1	0	13.3
2	0.5	12.5
3	1.0	12.0
4	1.5	10.5
5	2.0	9.5
6	2.5	8.2
7	4.7	4.2
8	5.0	4.5
9	5.5	4.3
10	6.0	5.0
11	7.0	5.4
12	7.5	5.6
13	9.5	6.7
14	10.0	7.5
15	10.5	7.9
16	11.0	9.0
17	11.5	9.6
18	12.0	10.5

★ Calculations

strength of strong acid →

- Volume of NaOH (V_2) = 4.7 ml (end point I)
- Normality of NaOH (N_2) = 0.1 N
- Volume of mixture (V_1) = 10 ml
- Normality of HCl (N_1) = $\frac{N_2 \times V_2}{V_1} = \frac{0.1 \times 4.7}{10} = 0.047 \text{ N}$
- Strength of HCl = $(0.047 \times 36.5) \text{ g/L} = 1.716 \text{ g/L}$

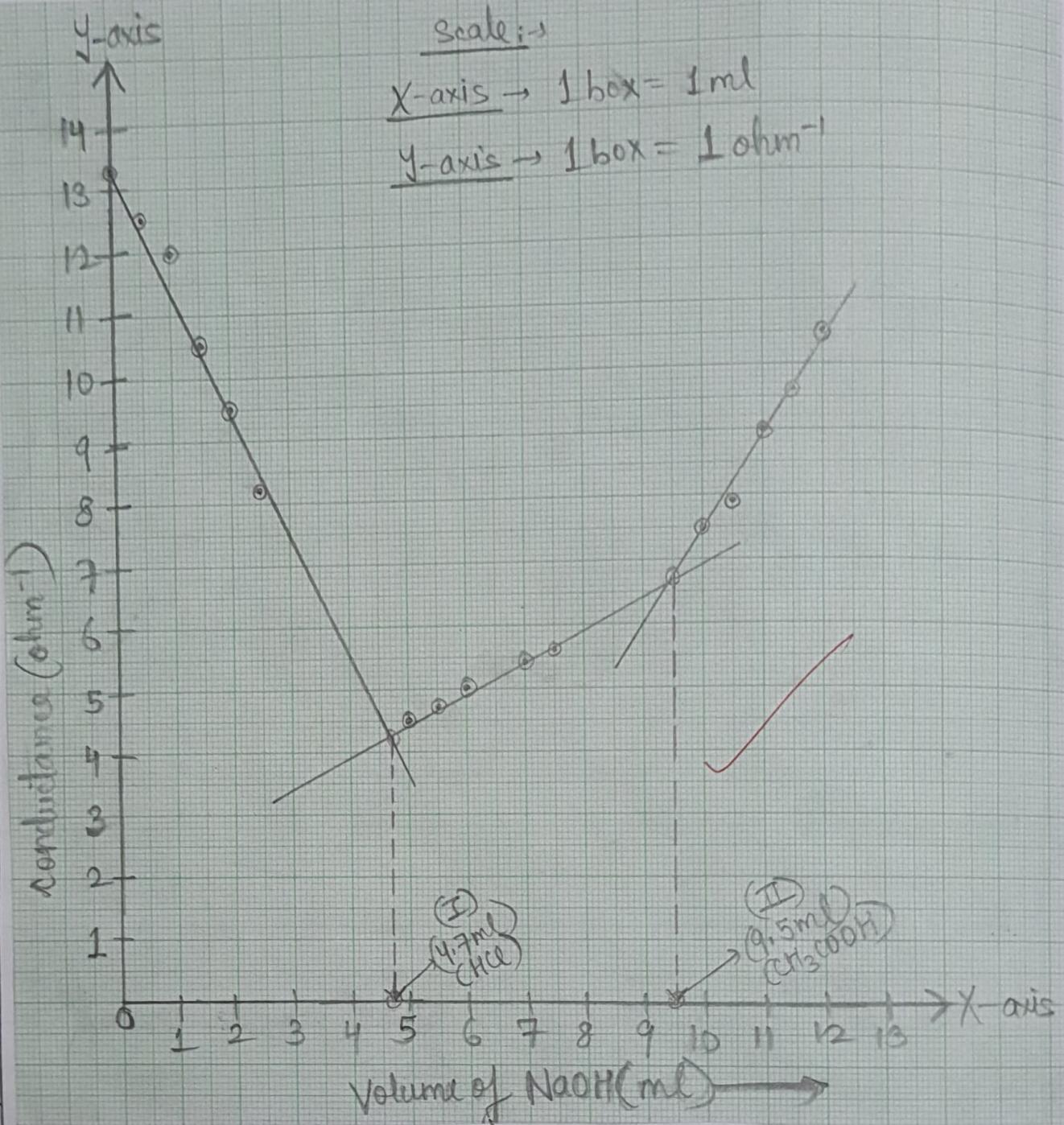
neutralisation. After complete neutralization, the amount of acid present in the given mixture is determined based on the volume of NaOH consumed. Volume of base consumed for strong acid and weak acid are determined by plotting a graph b/w conductance and volume of base added, where first end point corresponds to strong acid and second end point corresponds to weak acid.



Result:-

- The strength of HCl present in the whole of given solution = ~~0.047 N~~ 1.716 g/L X
- The strength of CH_3COOH present in the whole of given solution = ~~2.88 g/L~~ ✓
amount

R:



* Calculations (con)

Strength of weak acid:-

$$\text{Volume of mixture (V)}_1 = 10 \text{ ml}$$

$$\text{Volume of NaOH (V}_3) = (9.5 - 4.7) \text{ ml} \quad [2^{\text{nd}} \text{ end pt} - V_2] \\ = 4.8 \text{ ml}$$

$$\text{Normality of NaOH (N}_2) = 0.1 \text{ N}$$

$$\text{Normality of CH}_3\text{COOH (N}_3) = \frac{N_2 \times V_3}{V_1} = \frac{(0.1)(4.8)}{10} = 0.048 \text{ N}$$

$$\text{Strength of CH}_3\text{COOH} = [(0.048)(60)] \text{ g/L} = 2.88 \text{ g/L}$$

Experiment - No - 6

(Determination of strength of an acid using
pH meter)

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

Apparatus Reqd: → pH meter, glass electrode, beakers, standard flask.

Reagents Reqd: → 0.1 N NaOH solⁿ, unknown strength of HCl solⁿ, buffer solⁿ (pH = 7 and pH = 1).

Principle: → When an alkali is added to an acid solⁿ, the pH of the solⁿ increases slowly but at vicinity of the end pt., the rate of change of pH of the solⁿ is very rapid. A plot is drawn b/w volume of the alkali added and pH of the solⁿ. The sharp break in the curve gives the equivalence point from which the strength can be evaluated, using normality eqⁿ.

$$\text{pH} = -\log [\text{H}^+]$$

The pH of the solⁿ is defined as the -ve logarithm of the H⁺ ion concentration.

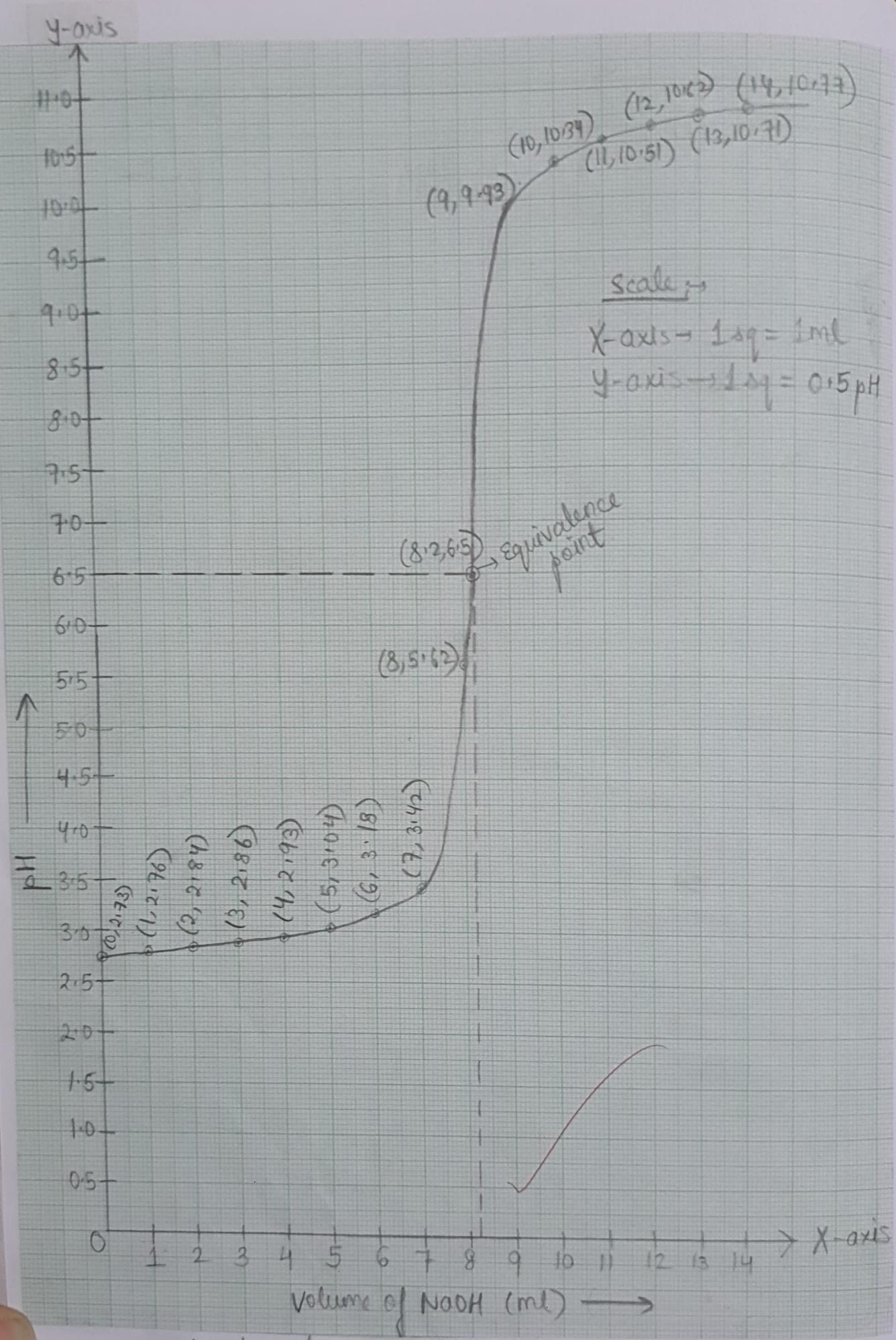
Table-1 :- Pilot titration b/w HCl v/s NaOH

S.NO	Volume of NaOH(V) ml	pH
1	0	2.73
2	1	2.76
3	2	2.84
4	3	2.86
5	4	2.93
6	5	3.04
7	6	3.18
8	7	3.42
9	8	5.62
10	9	9.93
11	10	10.34
12	11	10.51
13	12	10.62
14	13	10.71
15	14	10.77



Procedure: → Standardize the meter with acidic buffer. Wash the electrode with distilled water and dry. Take 10 ml HCl solⁿ in a beaker and dip electrodes completely in it. Note the pH of the acid solⁿ. Add to the solⁿ 1 ml of 0.1 N NaOH solⁿ from burette, stir well and note down the pH of the solⁿ. Similarly go on adding the alkali up to say 9-10 ml, when pH changes occur rapidly (equivalence pt), add alkali in fraction (0.2 ml).

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 solⁿ. The given acid is made upto 100 ml using distilled water. 10 ml of this made-up solⁿ is pipetted out into a 250 ml clean beaker and 100 ml of distilled water is added, so that the glass rod as well as the reference electrode is completely dipped. Note the pH of the pure acid solⁿ. Fill the burette with standard NaOH solⁿ and run down into the beaker in small increments (0.5 ml). Stir the solution well using glass rod. Note down the pH of every successive addition. Continue the titration



graph based on Pilot Titration.

fill beyond the neutralisation point as indicated by an abrupt change in pH (at least 30 increments). Plot a graph b/w 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 solⁿ close to the end pt. (1.0 ml on either side of the range) and measure the pH of every addition. Plot a fair graph b/w volume of NaOH against $\Delta \text{pH} / \Delta V$. find out the exact end point from the fair graph. The peak point of the curve from the fair graph gives the equivalence point.

P.T.O. →

Table - 2 :- Faj's titration b/w HCl v/c NaOH

S.NO	Volume of NaOH (mL)	pH	ΔpH	ΔV	$\Delta pH / \Delta V$
1	0	2.70			
2	1	2.73	0.03	1	0.03
3	2	2.80	0.07	1	0.07
4	3	2.83	0.03	1	0.03
5	4	2.90	0.07	1	0.07
6	5	3.00	0.10	1	0.10
7	6	3.17	0.17	1	0.17
8	6.2	3.20	0.03	0.2	0.15
9	6.4	3.24	0.04	0.2	0.20
10	6.6	3.28	0.04	0.2	0.20
11	6.8	3.36	0.08	0.2	0.40
12	7.0	3.43	0.07	0.2	0.35
13	7.2	3.51	0.08	0.2	0.40
14	7.4	3.63	0.12	0.2	0.60
15	7.6	3.75	0.12	0.2	0.60
16	7.8	4.10	0.35	0.2	1.75
17	8.0	6.20	2.10	0.2	10.50
18	8.2	8.56	2.36	0.2	11.80
19	8.4	9.12	0.56	0.2	2.80
20	8.6	9.53	0.41	0.2	2.05
21	8.8	9.69	0.16	0.2	0.80
22	9.0	9.88	0.19	0.2	0.95
23	9.2	9.98	0.10	0.2	0.50
24	9.4	10.11	0.13	0.2	0.65
25	9.6	10.16	0.05	0.2	0.25
26	9.8	10.25	0.09	0.2	0.45
27	10.0	10.31	0.06	0.2	0.30
28	10.2	10.35	0.04	0.2	0.20
29	10.4	10.38	0.03	0.2	0.15
30	10.6	10.41	0.03	0.2	0.15
31	10.8	10.46	0.05	0.2	0.25
32	11.0	10.48	0.02	0.2	0.10

Calculations:-

Volume of NaOH (V_1) = 8.2 ml (from FAIR graph)

Strength of NaOH (N_1) = 0.1 N

Volume of HCl (V_2) = 10 ml

Strength of HCl (N_2) = $\frac{N_1 \times V_1}{V_2}$

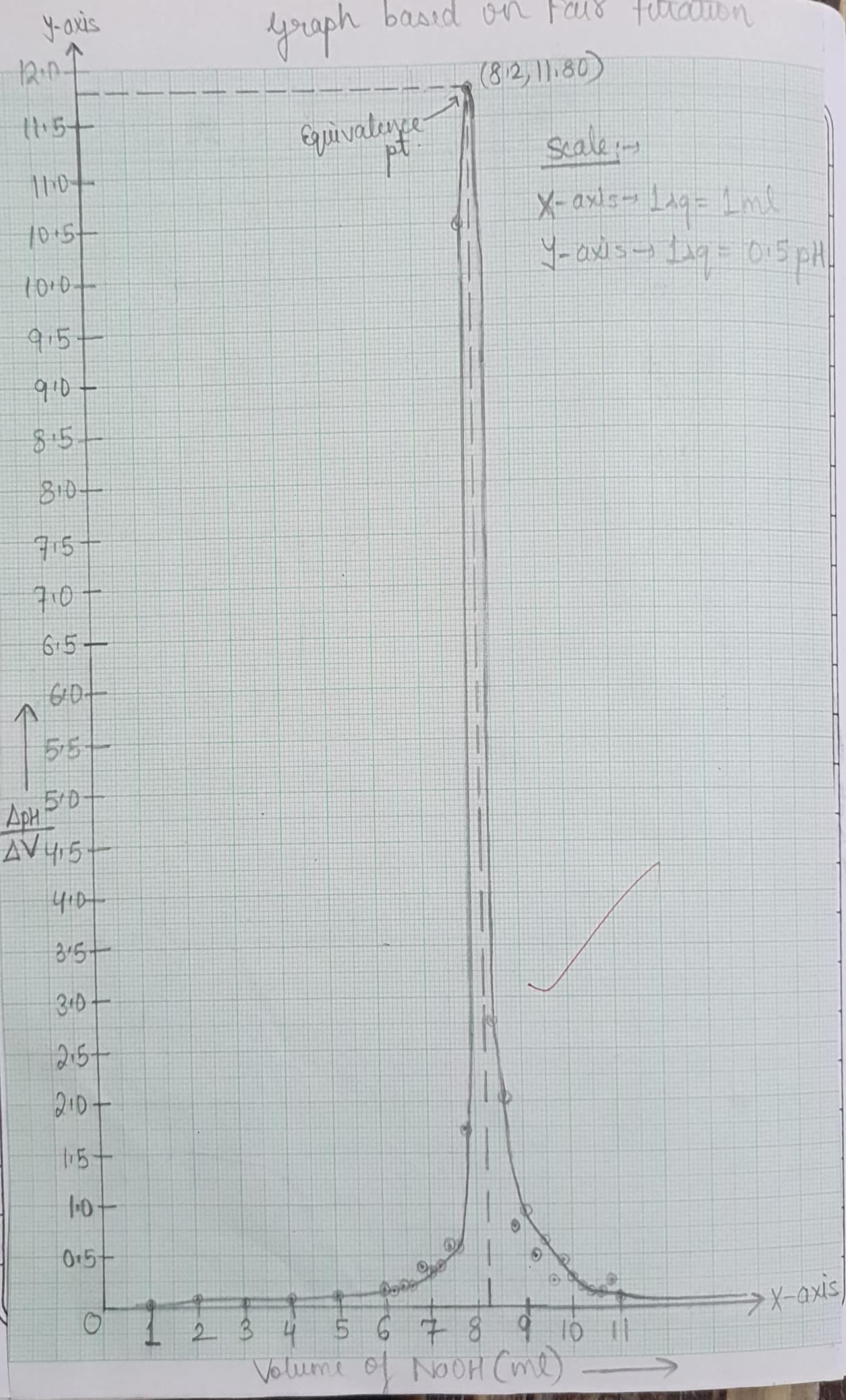
$$= \frac{(0.1)(8.2)}{10}$$

$$= 0.082 N$$

★ Result → The strength of given HCl soln is
0.082 N

Q
✓
0.082 N

Graph based on Fair titration



Experiment No - 7

(Estimation of Iron by Potentiometric Titration)

- ★ Aim → To estimate the amount of Fe^{2+} ion present in the given solution!
- ★ Apparatus Req'd → Potentiometer assembly, 25 ml burette, 10 ml pipette, 250 ml beakers, standard flask, calomel and platinum electrodes.
- ★ Reagents Req'd → Ferrous ammonium sulphate (FAS), dil. H_2SO_4 , std. $\text{K}_2\text{Cr}_2\text{O}_7$.
- ★ Principle → Measurement of EMF of an electrochemical cell using potentiometer and change in EMF due to the chemical (redox) rxn 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 oxidising to Fe^{3+} as $\text{K}_2\text{Cr}_2\text{O}_7$ is progressively added. Pt electrode, which is kept in contact with a mixture of Fe^{2+} and Fe^{3+} ions, act as a redox electrode (indicating the redox rxn). The reduction potential of this single electrode

Table-1:- FAS v/s $K_2Cr_2O_7$ (Pilot Titration)

S.NO	volume of $K_2Cr_2O_7$ (ml)	EMF (volts)	ΔE (Volts)
0		0	-
1		40	40
2		70	30
3		95	25
4		130	35
5		340	210
6		375	35
7		390	15
8		410	20
9		422	12
10		431	9

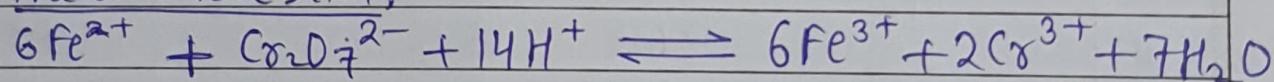
100 ml
10 ml
1 ml
1/10 ml

depends on the ratio of $[Fe^{2+}/Fe^{3+}]$ initially.

During the titration of Fe in H_2SO_4 medium with $K_2Cr_2O_7$, this ratio varies to a little extent at the beginning & suddenly near the end point. After the end pt, the ratio change is very little. It can be noted that there is a sudden change in the ratio of $[Fe^{3+}/Fe^{2+}]$ as the equivalence pt. is reached. This causes a sudden increase in the EMF of the cell at equivalence pt.

The cell set up: $Hg | HgCl_2(s), KCl(1N) || Fe^{2+}/Fe^{3+}, Pt$

The chemical rxn:

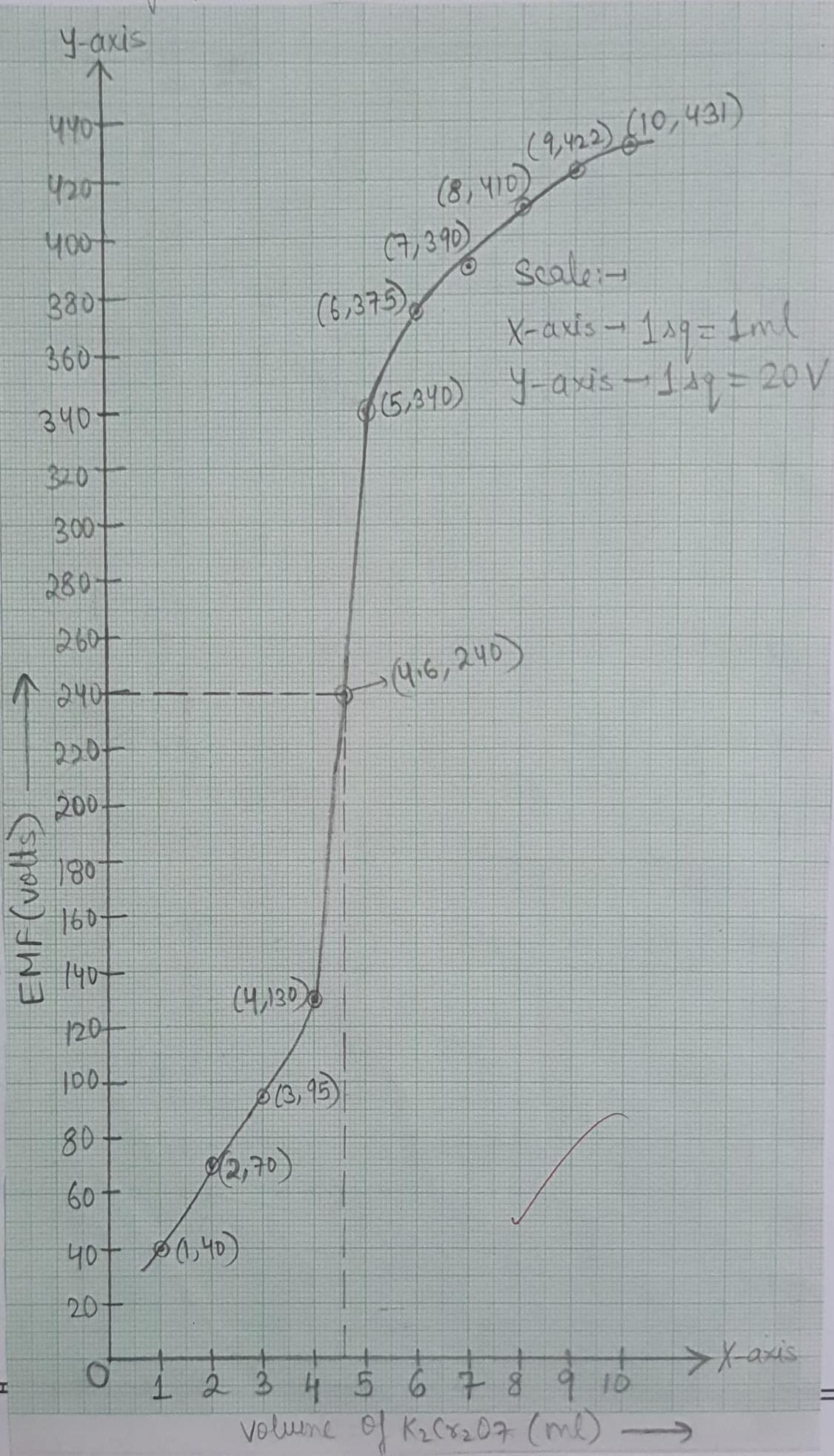


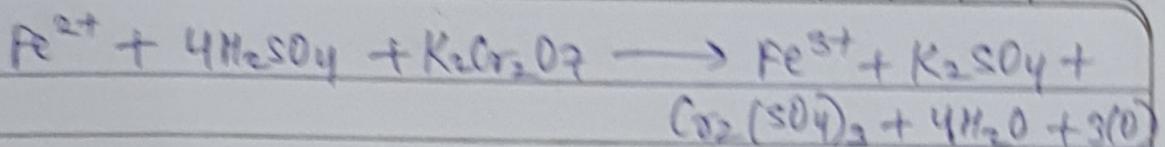
Potentiometric titration is the titration in which potentiometric measurements are carried out in order to fix the end point. In this method the interest is with the change in electrode potential, rather than with an accurate value

for the electrode potential in a given soln.

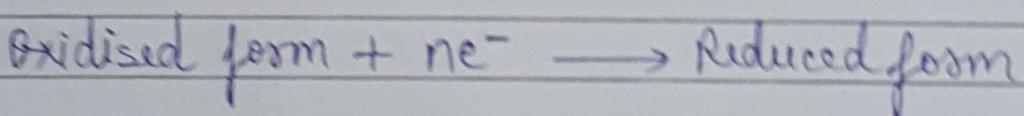
In a potentiometric titration, the change in EMF occurs most rapidly in the neighbourhood of the end pt. The $Fe(II) - K_2Cr_2O_7$ redox system is represented as -

graph based on Pilot Titration.





The determining factor is the ratio of the concentrations of the oxidised and the reduced form of the iron species. For the ox/red,



The potential E acquired by the indicator electrode at 25°C is given by,

$$E = E^{\circ} + \frac{0.0591}{n} \log \frac{[\text{Ox}]}{[\text{Red}]}$$

where E° is the std. redⁿ potential of the system. Thus, the potential of the immersed electrode is controlled by the ratio of these concentrations. During redox rxns, the potential changes more rapidly at the vicinity of the end pt. The indicator electrode is usually a bright platinum wire or foil, the oxidising agent is taken in the burette. The cell can be represented as -

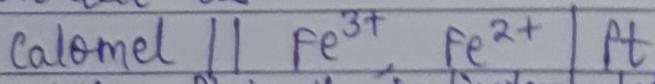


Table-2: FAS v/s $K_2Cr_2O_7$ (Fair Titration)

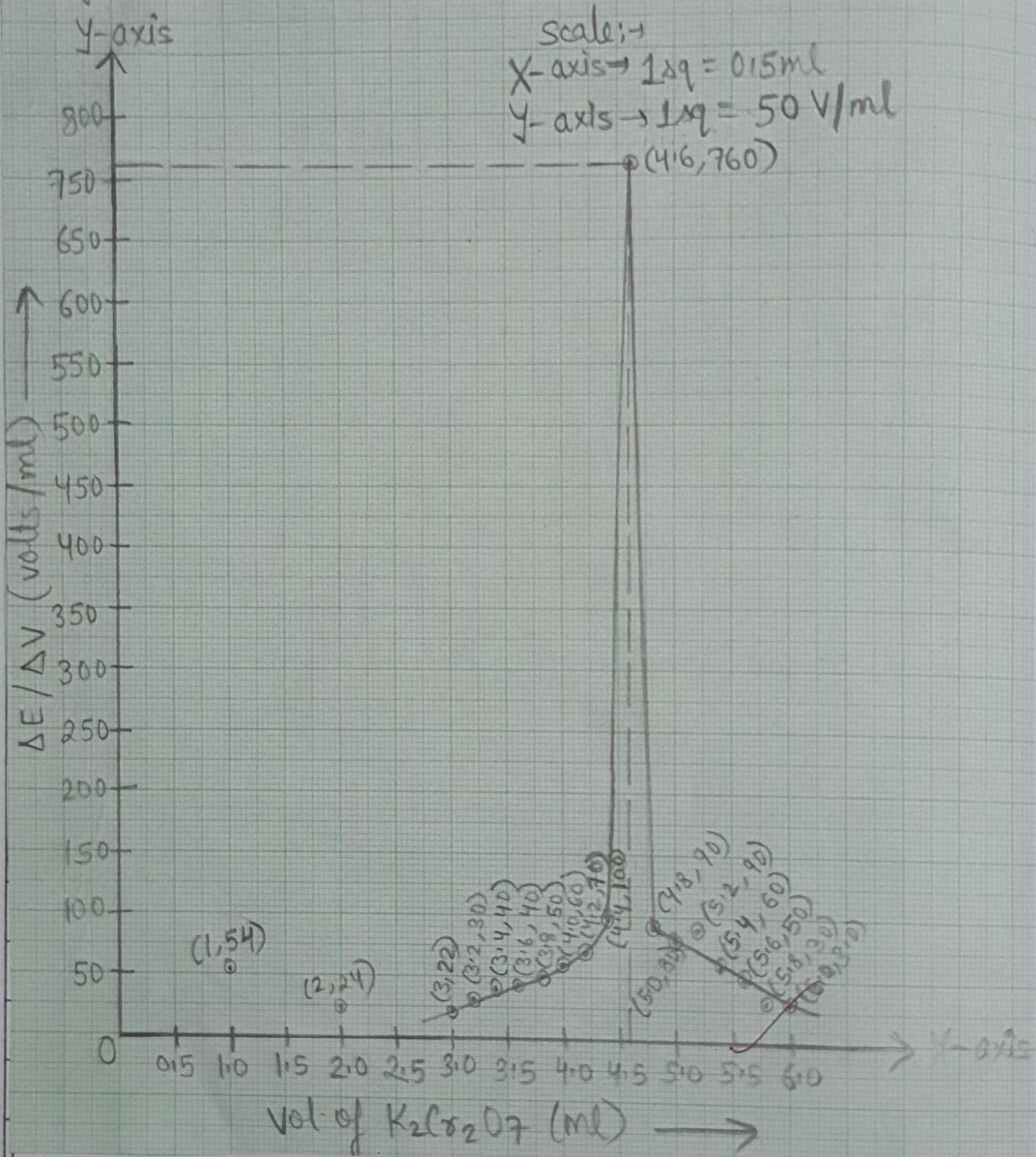
S.No	Vol. of $K_2Cr_2O_7$ (mL)	EMF (volts)	ΔE (volts)	ΔV (mL)	$\Delta E / \Delta V$ (volts/mL)
1	0	0	—	—	—
2	1.0	54	54	1.0	54
3	2.0	78	24	1.0	24
4	3.0	100	22	1.0	22
5	3.2	106	6	0.2	30
6	3.4	114	8	0.2	40
7	3.6	122	8	0.2	40
8	3.8	132	10	0.2	50
9	4.0	144	12	0.2	60
10	4.2	158	14	0.2	70
11	4.4	178	20	0.2	760
12	4.6	330	152	0.2	90
13	4.8	348	18	0.2	80
14	5.0	364	16	0.2	90
15	5.2	382	18	0.2	60
16	5.4	394	12	0.2	50
17	5.6	404	10	0.2	30
18	5.8	410	6	0.2	30
19	6.0	416	6	0.2	30

★ Procedure →

□ Pilot Titration : →

- The given ferrous ion solⁿ is made up to a known volume (say 100ml) in a SMF following the standard procedure with usual precautions.
- Exactly 10 ml of the made up Fe²⁺ solⁿ is pipette out into a clean 100ml beaker. About 10 ml of dilute H₂SO₄ and 100ml distilled water are added to it.
- A Pt electrode is dipped into this solⁿ and coupled with the std. calomel electrode. The resultant cell is then incorporated into the potentiometric circuit.
- Std. K₂C₂O₇ solⁿ, which is taken in a burette, is added in installments of 1ml into the beaker and the cell EMF is measured after each addition by proper mixing.
- The process is continued till and also well beyond the neutralisation pt. as indicated by an abrupt change in the EMF.

graph based on Far's Titration



Fair Titration:-

- Note the volume of $K_2Cr_2O_7$ solⁿ reqd. for complete oxidⁿ of Fe^{2+} solⁿ from the plot of emf v/s the volume of std. $K_2Cr_2O_7$ solⁿ added. The range at which the end-pt. lies may be evaluated.
- Calculate the normality of given Fe^{2+} solⁿ using the formula $N_1V_1 = N_2V_2$.
- One more similar titration is performed by adding 0.1 ml portions of std. $K_2Cr_2O_7$ solⁿ close to the end pt. (1 ml on either side of the range).
- graph is plotted:-
 (a) EMF v/s Volume of $K_2Cr_2O_7$ addition
 (b) $\Delta E / \Delta V$ v/s Volume of $K_2Cr_2O_7$ addition

The exact end pt. can be determined from the plot of $\Delta E / \Delta V$ v/s volume of std. $K_2Cr_2O_7$ solⁿ.

Atomic wt. of Fe is 55.85.

Result:-

The amount of iron present in the whole of the given solⁿ is 0.2576 g. *(Ans)*

* Calculations

Volume of pipette soln (FAS) (V_1) = 10 ml

Volume of $K_2Cr_2O_7$ (V_2) = 4.6 ml

Normality of $K_2Cr_2O_7$ (N_2) = 0.1 N

$$\begin{aligned} \text{Normality of FAS (N)} &= \frac{(N_2 V_2)}{V_1} \\ &= \frac{(0.1)(4.6)}{10} \\ &= 0.046 \text{ N} \end{aligned}$$

$$\begin{aligned} \text{Amount of } Fe^{2+} (\text{g/L}) &= \text{Eq. wt} \times \text{Normality of } Fe^{2+} \\ &= (28 \times 0.046) \text{ g/L} \\ &= 1.288 \text{ g/L} \end{aligned}$$

$$\begin{aligned} \text{Amount of } Fe^{2+} \text{ in 100 ml} &= \text{Normality of } Fe^{2+} \times \frac{56}{10} \\ &= 0.046 \times 56 \times 0.1 \\ &= 0.2576 \text{ g / 100ml} \end{aligned}$$