

Aim - To verify freundlich and langmuir adsorption isotherms for the adsorption of acetic acid on activated charcoal.

Theory :-

Adsorption is a surface phenomenon in which molecules of a substance get accumulated on the surface of another substance. In this experiment, acetic acid is adsorbed from its aqueous solution on the surface of activated charcoal.

The substance which get adsorbed is called adsorbate and the surface on which adsorption occurs is called adsorbent.

Two important adsorption isotherms are :-

1) Freundlich adsorption isotherm

It is represented by the equation

$$\frac{x}{m} = Kc^{1/n}$$

Taking logarithm

$$\log x/m = \log k + 1/n \log c$$

where :-
 x = Amount of acetic acid adsorbed
 m = Mass of charcoal
 c = Equilibrium concentration
 k and n = constants

A graph between $\log x/m$ and $\log c$ gives a straight line

2. Langmuir Adsorption Isotherm

It is represented by

$$\frac{x}{m} = \frac{abc}{1 + ac}$$

on rearrangement

$$\frac{c}{x/m} = \frac{1}{ab} + \frac{1}{b} c$$

A graph between $c/(x/m)$ and c gives a straight line, verifying Langmuir isotherm.

Requirements -

Six stoppered reagent bottles (250 mL)

Calculation

Amount of acetic acid adsorbed

$$x = \frac{(C_0 - C_e) \times V \times \text{Eq. wt.}}{1000}$$

Where, C_0 = Initial Concent.

C_e = Equilibrium conc.

V = 50 mL

Equivalent weight of acetic acid = 60

m = 2 gm

Bottle No. 1

Given :- $C_0 = 0.250 \text{ mol L}^{-1}$

$C_e = 0.184 \text{ mol L}^{-1}$

$V = 50 \text{ mL}$

Equivalent weight of acetic acid = 60

Calculation of x

$$x = \frac{(C_0 - C_e) \times V \times \text{Eq. wt.}}{1000}$$

$$= \frac{(0.250 - 0.184) \times 50 \times 60}{1000}$$

$$x = 0.198 \text{ g}$$

Calculation of x/m

$$x/m = 0.198/2$$

$$= 0.099$$

Langmuir term

$$C_e/(x/m) = 0.184/0.099 = 1.86$$

Bottle No. 2

$C_0 = 0.200 \text{ mol L}^{-1}$

$C_e = 0.152 \text{ mol L}^{-1}$

Calculation of x

$$x = \frac{(0.200 - 0.152) \times 50 \times 60}{1000}$$

$$x = 0.144 \text{ g}$$

Volumetric flasks, burette, pipette, conical flasks, analytical balance, finely powdered activated charcoal, 0.25M acetic acid solution, 0.1M NaOH solution, phenolphthalein indicator, filter paper, and funnels.

Observations

- Temperature of the experiment = 25
- Mass of activated charcoal in each bottle (m) = 2.0g
- Total Volume of solution in each bottle (V) = 50ml
- Molarity of standard base (NaOH) = 0.1M
- Equivalent weight of acetic acid = 60.05 g/mol

Table

S.No.	Weight of charcoal (g)	Vol ^m of acetic acid (ml)	Vol ^m of water (ml)	Initial conc. (C ₀) (mol L ⁻¹)	Vol ^m of filtrate (ml)	NaOH used (ml)	Equil. Conc. (C _e) (mol L ⁻¹)	Acetic acid Adsorbed (x) (g)	(x/m)	log (x/m)	log (C _e) (mol L ⁻¹)	log (C ₀) (mol L ⁻¹)
1	2.0	50	0	0.250	5	9.2	0.184	0.198	0.099	1.004	0.959	1.86
2	2.0	40	10	0.200	5	7.6	0.152	0.144	0.072	1.143	0.735	2.11
3	2.0	30	20	0.150	10	5.5	0.110	0.060	0.030	1.523	0.818	3.67
4	2.0	20	30	0.100	10	3.8	0.076	0.036	0.018	1.745	1.119	4.22
5	2.0	10	40	0.050	20	2.1	0.042	0.012	0.006	2.222	1.377	7.00
6	2.0	5	45	0.025	20	1.0	0.020	0.003	0.0015	2.824	1.699	13.33

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Calculation of x/m

$$x/m = 0.144/2 = 0.072$$

Log values

$$\log(x/m) = \log(0.072) = -1.143$$

$$\log C_e = \log(0.152) = -0.818$$

Langmuir term

$$C_e/(x/m) = 0.152/0.072 = 2.11$$

Bottle No. 3

$$C_0 = 0.150 \text{ mol L}^{-1}$$

$$C_e = 0.110 \text{ mol L}^{-1}$$

Calculation of x

$$x = [(0.150 - 0.110) \times 50 \times 60] / 1000$$

$$x = 0.060 \text{ g}$$

Calculation of x/m

$$x/m = 0.060/2 = 0.030$$

log values

$$\log(x/m) = \log(0.030) = -1.523$$

$$\log C_e = \log(0.110) = -0.959$$

Langmuir term

$$C_e/(x/m) = 0.110/0.030 = 3.67$$

Bottle No. 4

$$C_0 = 0.100 \text{ mol L}^{-1}$$

$$C_e = 0.076 \text{ mol L}^{-1}$$

Calculation of x

$$x = [(0.100 - 0.076) \times 50 \times 60] / 1000$$

$$x = 0.036 \text{ g}$$

Calculation of x/m

$$x/m = 0.036/2 = 0.018$$

log values

$$\log(x/m) = \log(0.018) = -1.745$$

$$\log C_e = \log(0.076) = -1.119$$

Langmuir term

$$C_e/(x/m) = 0.076/0.018 = 4.22$$

Procedure

- 1) Clean and dry 6 stoppered reagent bottle and label them from 1 to 6. Weigh exactly 2.0g of activated charcoal into each bottle.
- 2) Prepare varying concentrations of acetic acid by adding 5, 10, 20, 30, 40 and 5 ml of 0.25M acetic acid into bottles 1 to 6 respectively. Add distilled water (0, 10, 20, 30, 40 and 45 ml) to make the total volume 50 ml in each bottle.
- 3) Shake the bottle vigorously for 20 minutes and keep them in a water bath at room temp. for an hour with intermittent shaking to achieve equilibrium.
- 4) Filter the contents of each bottle through a dry filter paper into separate clean flasks. Reject the first 5 ml of the filtrate.
- 5) Pipette out a specific aliquot of the filtrate from each bottle (10 ml or 20 ml depending on concentration) and titrate against standard 0.1M NaOH using phenolphthalein as an indicator.

Bottle No. 5

$$C_0 = 0.050 \text{ mol L}^{-1}$$

$$C_e = 0.042 \text{ mol L}^{-1}$$

Calculation of x

$$x = [(0.050 - 0.042) \times 50 \times 60] / 1000$$

$$x = 0.012 \text{ g}$$

Calculation of x/m

$$x/m = 0.012 / 2 = 0.006$$

log value

$$\log(x/m) = \log(0.006) = -2.222$$

$$\log C_e = \log(0.042) = -1.377$$

Langmuir term

$$C_e / (x/m) = 0.042 / 0.006 = 7.00$$

Bottle No. 6

$$C_0 = 0.025 \text{ mol L}^{-1}$$

$$C_e = 0.020 \text{ mol L}^{-1}$$

Calculation of x

$$x = [(0.025 - 0.020) \times 50 \times 60] / 1000$$

$$x = 0.003 \text{ g}$$

Calculation of x/m

$$x/m = 0.003 / 2 = 0.0015$$

Log value

$$\log(x/m) = \log(0.0015) = -2.824$$

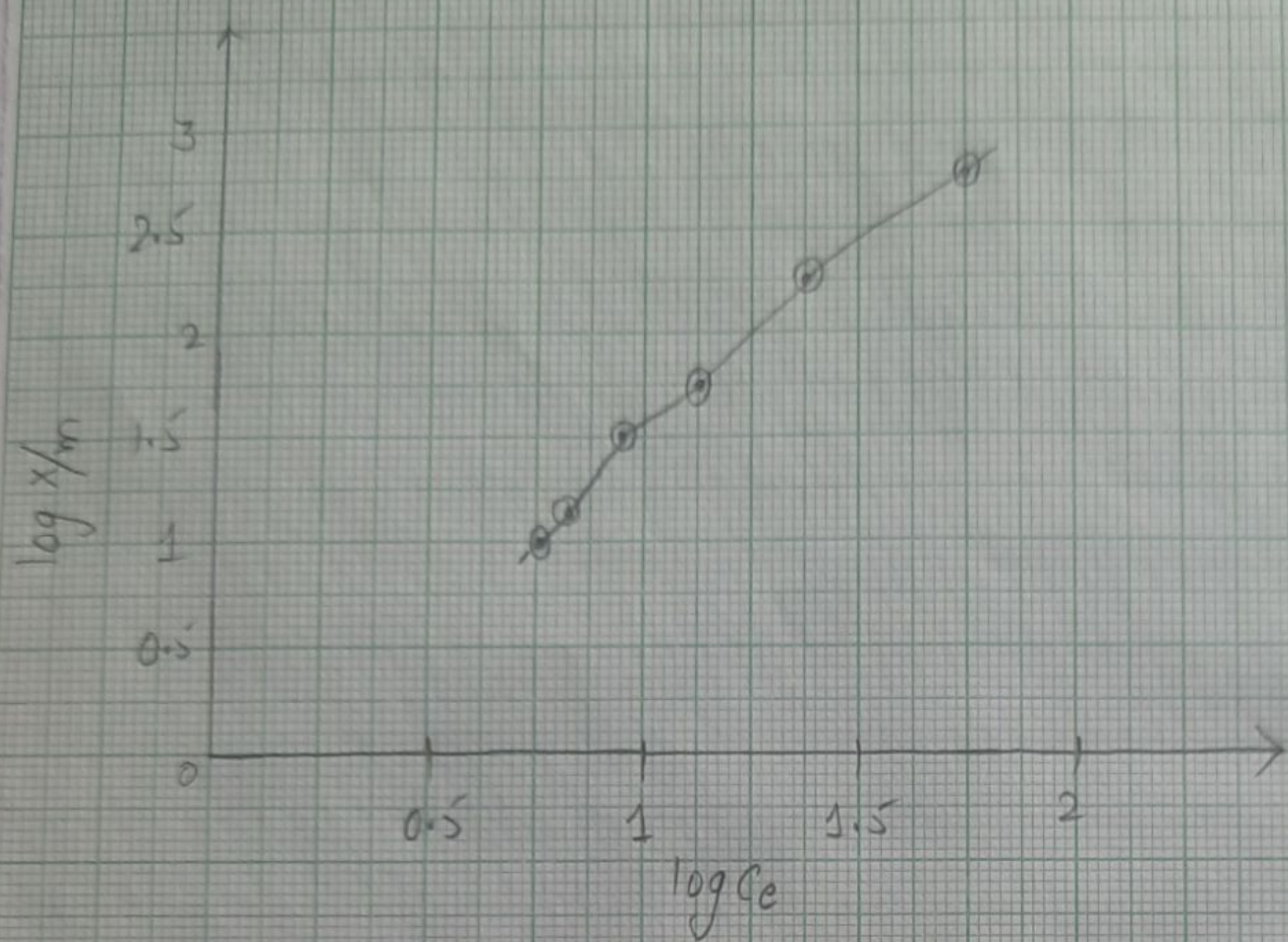
$$\log C_e = \log(0.020) = -1.699$$

Langmuir term

$$C_e / (x/m) = 0.020 / 0.0015 = 13.33$$

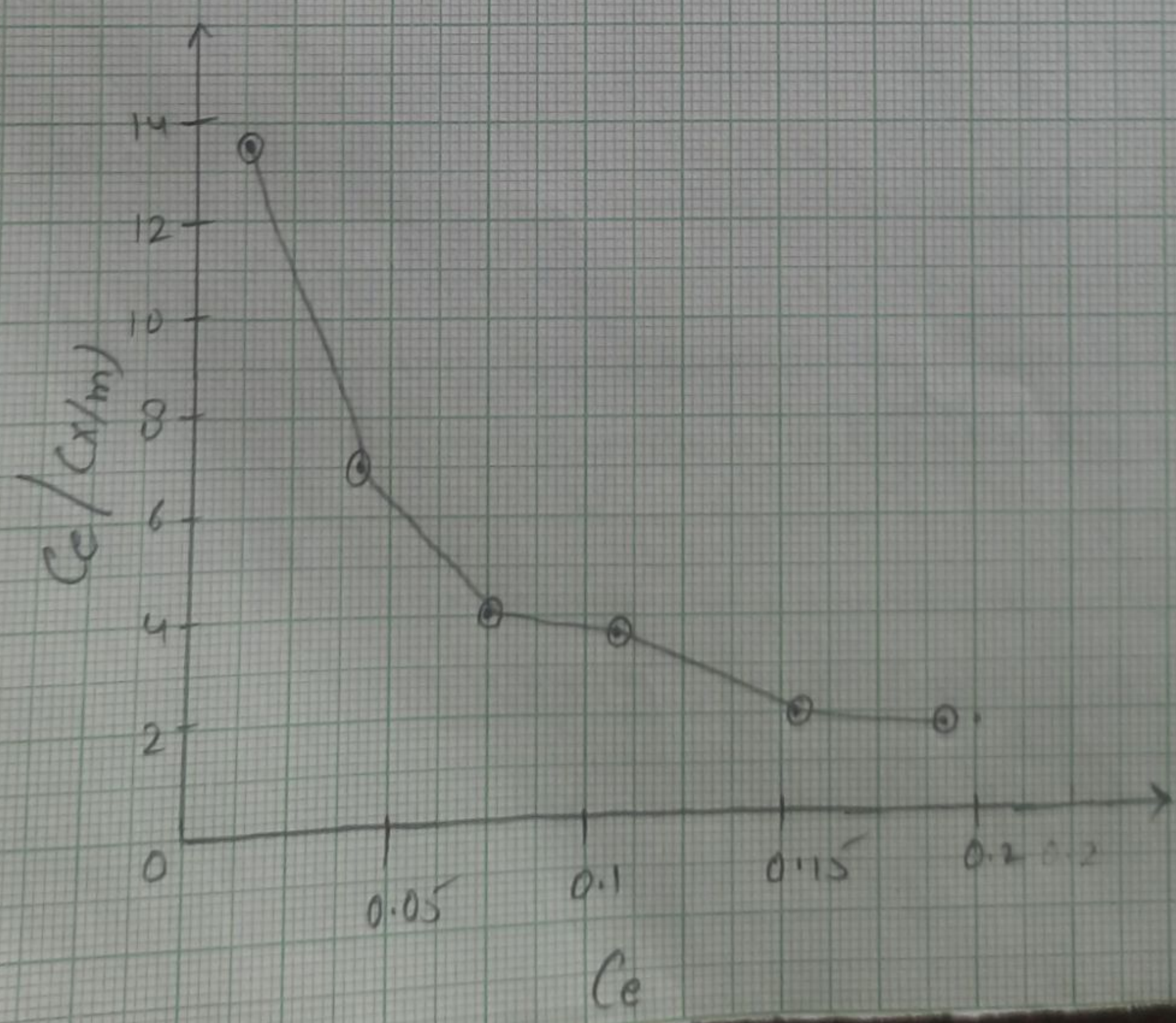
Freundlich Isotherm

(Ex. 1)



Graph plotted between $\log(x/m)$ and $\log C_e$ gives a straight line

Langmuir Isotherm



Result :-

The adsorption of acetic acid on activated charcoal obeys both freundlich adsorption isotherm and langmuir adsorption isotherm within the experimental concentration range.

Hence, the freundlich and langmuir adsorption isotherms were successfully verified.

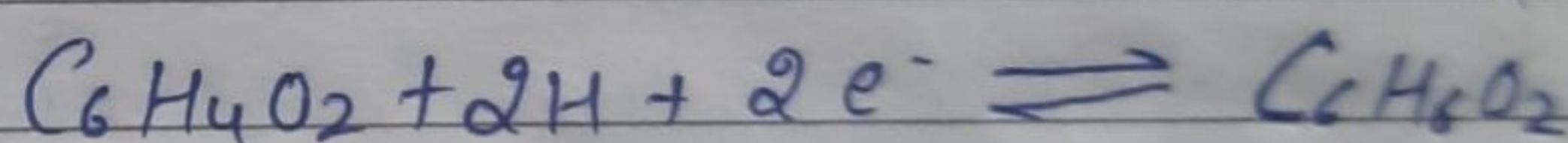
Aim:- To perform potentiometric titration of hydrochloric acid (HCl) against sodium hydroxide (NaOH) using quinhydrone electrode and determine the strength of NaOH solⁿ.

Principle :-

Potentiometric titration is based on the measurement of change in electrode potential during titration.

Quinhydrone is an equimolar mixture of quinone and hydroquinone.

Electrode reaction -



Acc. to Nernst equation.

$$E_{cell} = E^{\circ} + (0.0591/2) \log [H^+]^2 [Q] / [QH_2]$$

Since,

$$[Q] / [QH_2] = 1$$

Therefore

$$E = E^{\circ} + 0.0591 \log [H^+]$$

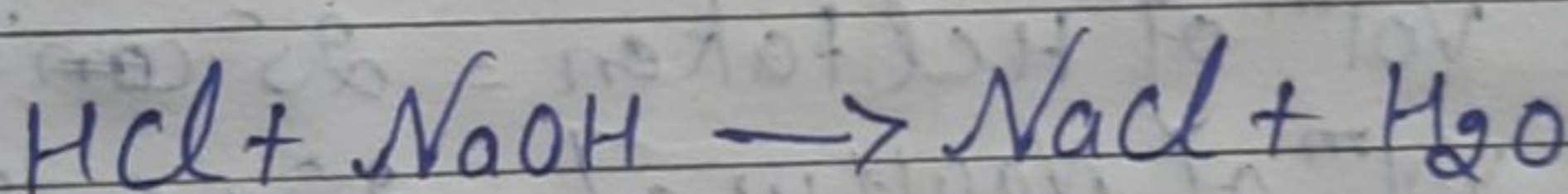
$$E = E^{\circ} - 0.0591 pH$$

For quinhydrone electrode

$$E = 0.6996 - 0.0591 pH$$

Thus, the potential of quinhydrone electrode depends upon pH of the solution

Neutralization reaction



During titration, change in H^+ ion concentration causes change in EMF. Near equivalence point, sharp change in potential is observed

Requirements

Apparatus

- Burette
- Volumetric flask
- Potentiometer
- Saturated calomel electrode
- Pipette
- Magnetic stirrer
- Beaker
- Platinum electrode
- Connecting wires

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1) Determination of Equivalence point

From the observation table, the maximum value of $\Delta E/\Delta V$ is obtained at 25.0 cm^3 of NaOH added. Therefore the equivalence point corresponds to 25.0 cm^3 of sodium hydroxide solution.

Hence,

Volume of NaOH used at equivalence point (V_2) = 25.0 cm^3

2) Calculation of Molarity of NaOH using the molarity relation

$$M_1 V_1 = M_2 V_2$$

where

M_1 = Molarity of HCl = 0.1 mol dm^{-3}

V_1 = Vol^m of HCl taken = 25.0 cm^3

V_2 = Vol^m of NaOH used = 25.0 cm^3

Substituting the values

$$0.1 \times 25.0 = M_2 \times 25.0$$

Therefore

$$M_2 = (0.1 \times 25.0) / 25.0$$

$$M_2 = 0.1 \text{ mol dm}^{-3}$$

3) Calculation of strength of NaOH solution

$$\text{Strength} = \text{Molarity} \times \text{Molar mass}$$

Chemicals

- Hydrochloric acid solution
- Sodium hydroxide solution
- Quinhydrone
- Distilled water

Observation table

S.No	Volume of NaOH Added v (ml)	EMF E (mV)	ΔV	ΔE	$\Delta E / \Delta V$
1	0.0	182	-	-	-
2	2.0	176	2.0	-6	-3.0
3	4.0	170	2.0	-6	-3.0
4	6.0	163	2.0	-7	-3.5
5	8.0	155	2.0	-8	-4.0
6	10.0	146	2.0	-9	-4.5
7	12.0	136	2.0	-10	-5.0
8	14.0	123	2.0	-13	-6.5
9	16.0	108	2.0	-15	-7.5
10	18.0	90	2.0	-18	-9.0
11	20.0	68	2.0	-22	-11.0
12	22.0	42	2.0	-26	-13.0
13	24.0	25	1.0	-17	-17.0
14	24.0	5	1.0	-20	-20.0
15	24.5 24.5	-18	0.5	-23	-46.0

$$\text{Molar mass of NaOH} = 23 + 16 + 1 \\ = 40 \text{ g mol}^{-1}$$

Therefore

$$\text{Strength of NaOH solution} = 0.1 \times 40 \\ = 4.0 \text{ g dm}^{-3}$$

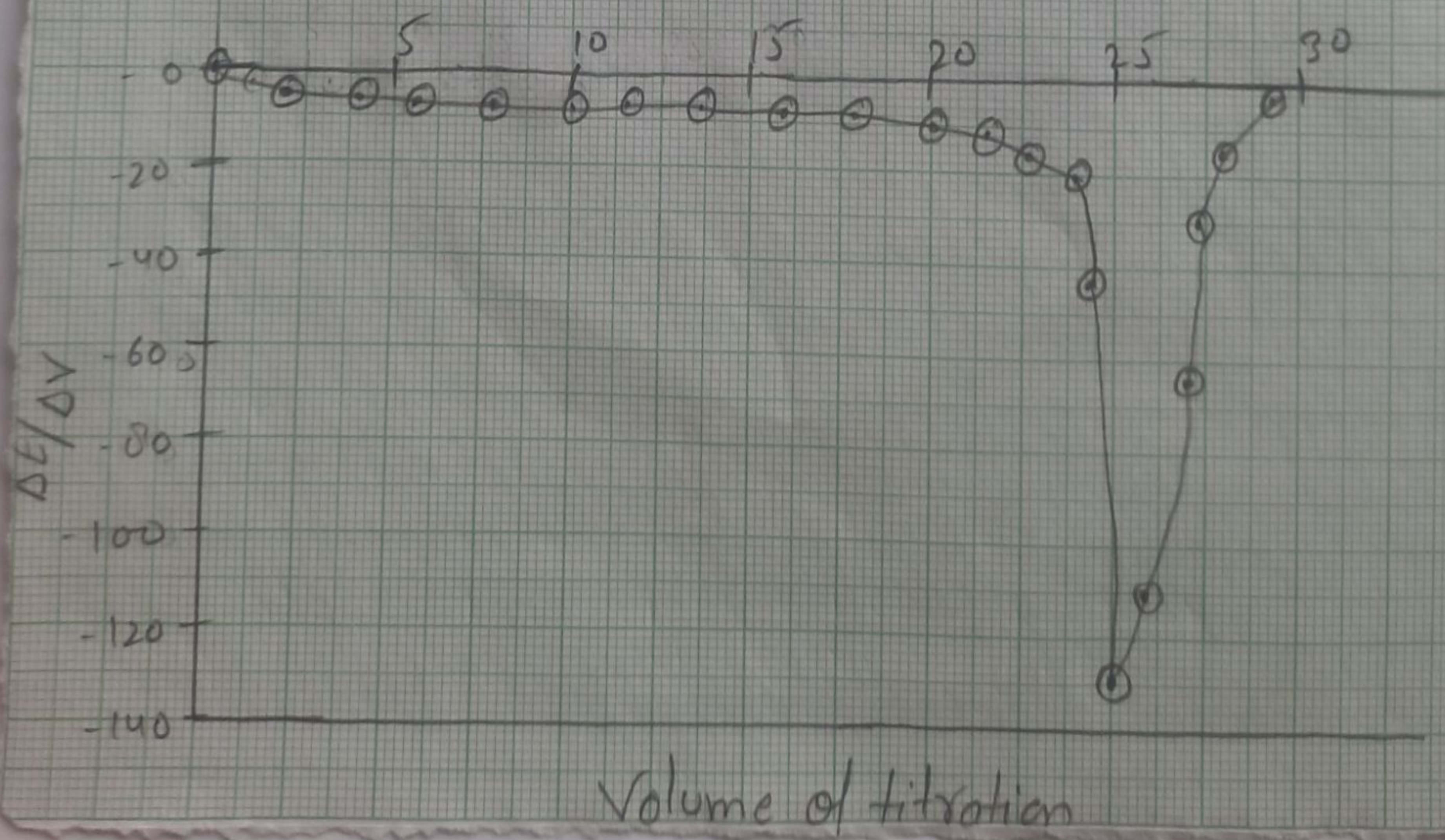
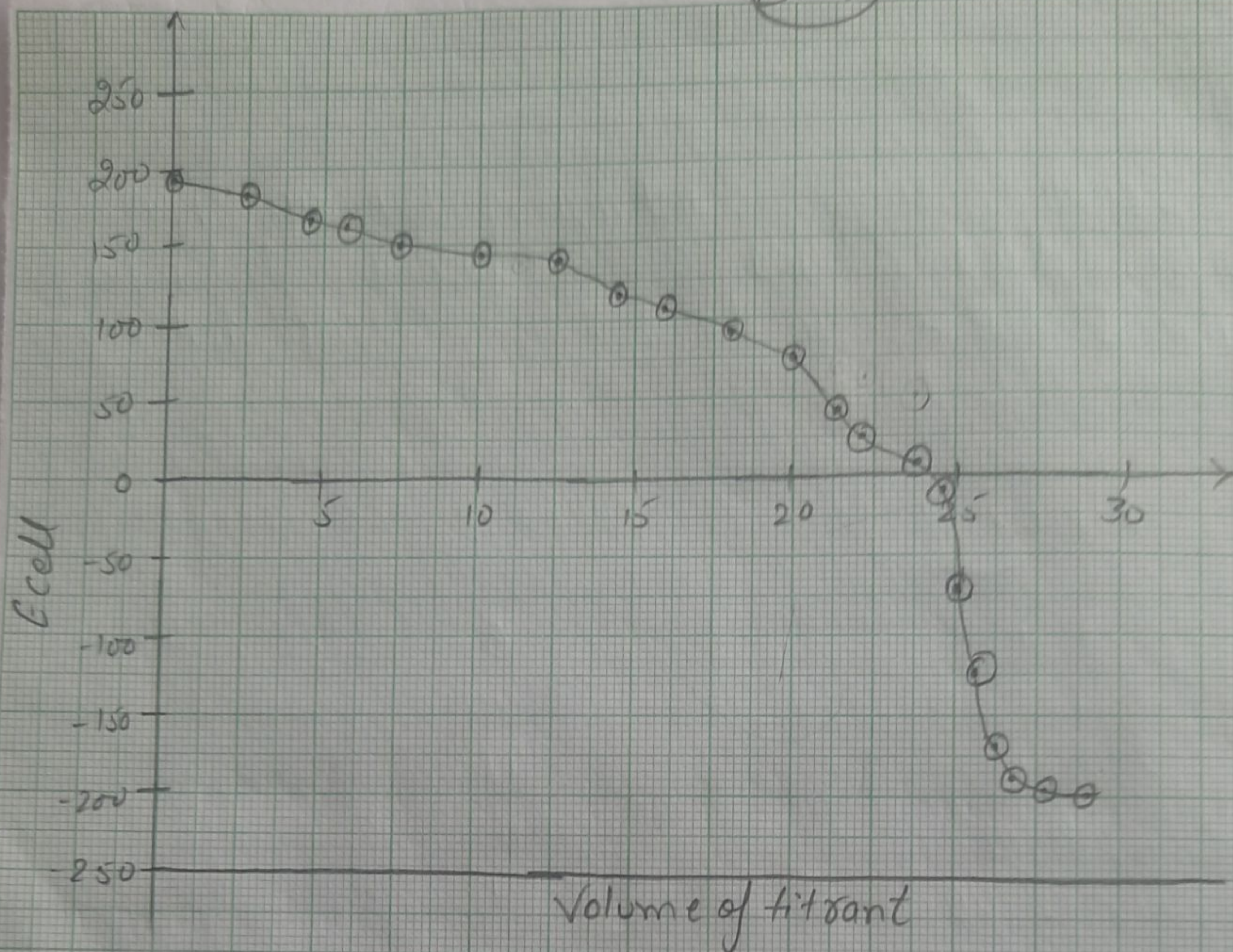
Volume of NaOH solution (cm ³)	Volume of HCl solution (cm ³)	Concentration of NaOH solution (g dm ⁻³)	Concentration of HCl solution (g dm ⁻³)	Volume of NaOH solution (dm ³)	Volume of HCl solution (dm ³)
0.0	0.0	0.0	0.0	0.0	0.0
0.5	0.5	0.5	0.5	0.5	0.5
1.0	1.0	1.0	1.0	1.0	1.0
1.5	1.5	1.5	1.5	1.5	1.5
2.0	2.0	2.0	2.0	2.0	2.0
2.5	2.5	2.5	2.5	2.5	2.5
3.0	3.0	3.0	3.0	3.0	3.0
3.5	3.5	3.5	3.5	3.5	3.5
4.0	4.0	4.0	4.0	4.0	4.0
4.5	4.5	4.5	4.5	4.5	4.5
5.0	5.0	5.0	5.0	5.0	5.0
5.5	5.5	5.5	5.5	5.5	5.5
6.0	6.0	6.0	6.0	6.0	6.0
6.5	6.5	6.5	6.5	6.5	6.5
7.0	7.0	7.0	7.0	7.0	7.0
7.5	7.5	7.5	7.5	7.5	7.5
8.0	8.0	8.0	8.0	8.0	8.0
8.5	8.5	8.5	8.5	8.5	8.5
9.0	9.0	9.0	9.0	9.0	9.0
9.5	9.5	9.5	9.5	9.5	9.5
10.0	10.0	10.0	10.0	10.0	10.0

16	25.0	-83	0.5	-65	-13.0
17	25.05	-140	0.5	-57	-114.0
18	26.0	-172	0.5	-32	-64.0
19	26.05	-188	0.5	-16	-32.0
20	27.0	-196	0.5	-8	-16.0
21	28.0	-202	1.0	-6	-6.0

Procedure

- Add a small pinch of quinhydrone powder to saturate the acid solution.
- Carry out the necessary electrical connections with the potentiometer or digital meter.
- Repeat the addition and note the steady E.M.F. after each increment.
- Dip the platinum indicator electrode and saturated Calomel electrode (SCE) into the beaker.
- Standardize the potentiometer using a standard cell before replacing it with the working cell setup.

(-X.2)



Result:-

The potentiometric titration of hydrochloric acid against sodium hydroxide was carried out Successfully.

Volume of NaOH required at equivalence point = 25.0 cm^3

Molarity of NaOH solution = 0.1 mol dm^{-3}

Strength of NaOH solution = 4.0 g dm^{-3}

Aim :- To perform potentiometric of acetic acid (CH_3COOH) against sodium hydroxide (NaOH) and determine the strength of sodium hydroxide solution.

Principle :-

Potentiometric titration is based on the measurement of change in electrode potential during titration.

In this experiment, acetic acid (weak acid) is titrated against sodium hydroxide (strong base) using quinhydrone electrode.

The neutralization reaction is



Quinhydrone electrode potential depends upon hydrogen ion concentration and is given by :-

$$E = E^\circ - 0.0591 \text{ pH}$$

During titration, H^+ ion concentration decreases gradually because acetic acid ionizes only partially.

Therefore, change in EMF is less sharp compared to strong acid - strong base titration.

At equivalence point, a noticeable change in potential is observed

Requirements

Apparatus

- Burette
- Potentiometer
- Magnetic stirrer
- Pipette
- Platinum electrode
- Connecting wires
- Beaker
- Saturated calomel electrode (SCE)

Chemicals

- Acetic acid solution
- Quinhydrone
- Sodium hydroxide solution
- Distilled water

Observation Table

Titration of CH_3COOH vs NaOH

Calculation

Given

$$M_1 = 0.1 \text{ mol dm}^{-3}$$

$$V_1 = 25.0 \text{ mL}$$

$$V_2 = 25.0 \text{ mL}$$

using relation

$$M_1 V_1 = M_2 V_2$$

Substituting values

$$0.1 \times 25.0 = M_2 \times 25.0$$

Therefore

$$M_2 = (0.1 \times 25.0) / 25.0$$

$$M_2 = 0.1 \text{ mol dm}^{-3}$$

Calculation of strength of NaOH

$$\text{Strength} = \text{Molarity} \times \text{Molar mass}$$

$$\text{Molar mass of NaOH} = 23 + 16 + 1 = 40 \text{ g mol}^{-1}$$

Therefore

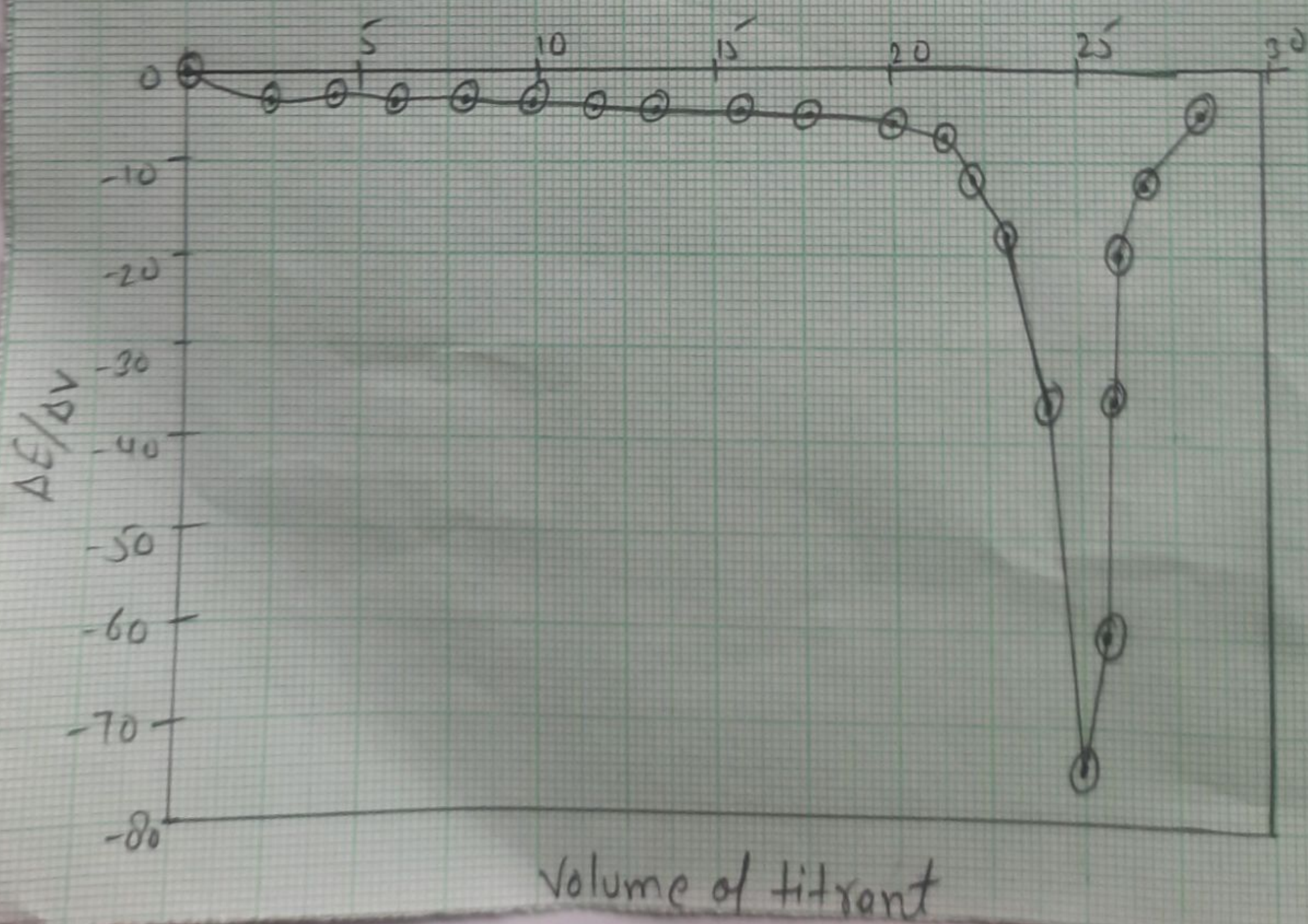
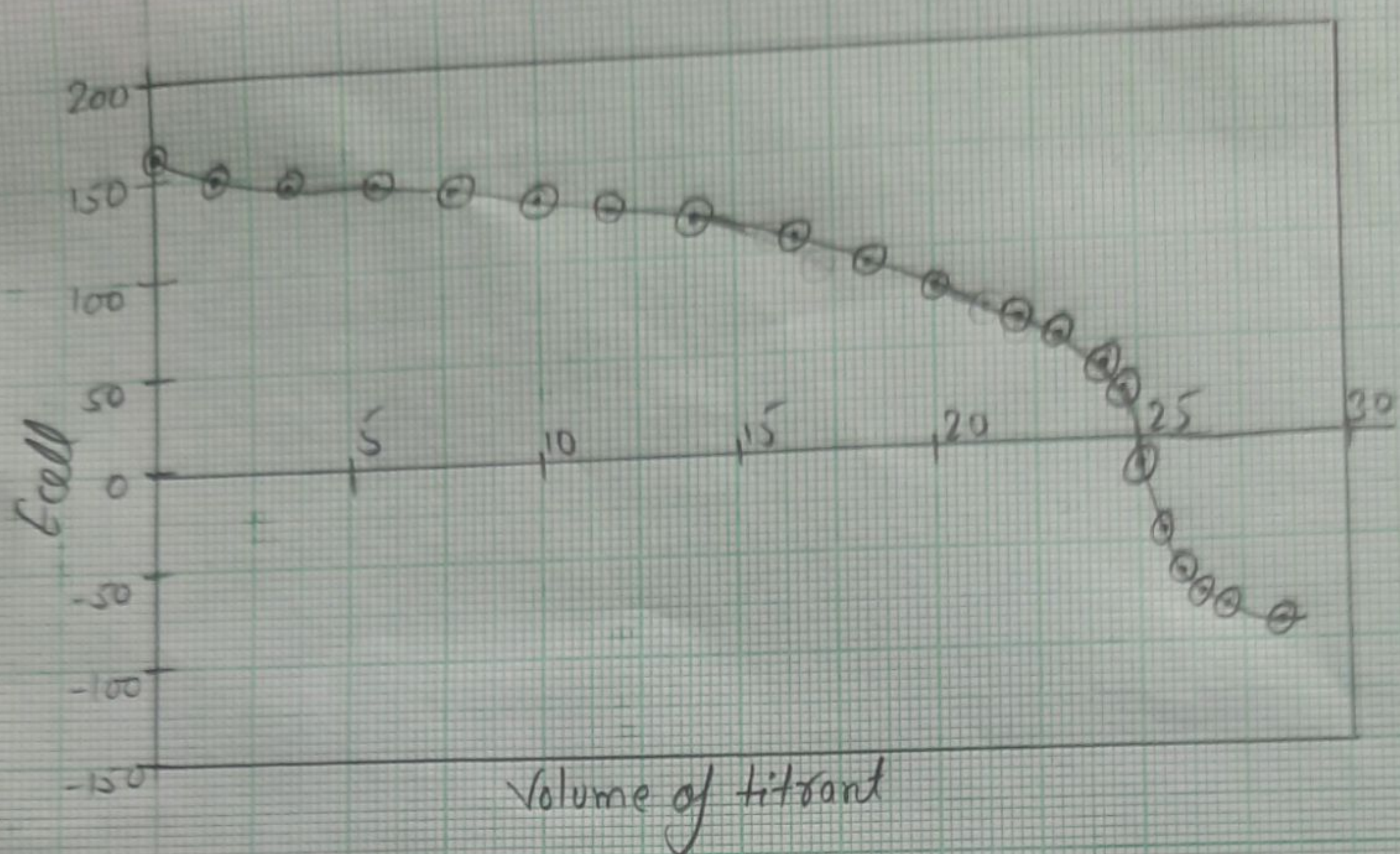
$$\text{Strength} = 0.1 \times 40 = 4.0 \text{ g dm}^{-3}$$

Expt. No. _____

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S.No	Volume of NaOH Added (mL)	EMF (E) (mV)	ΔV	ΔE	$\Delta E / \Delta V$
1	0.0	156	-	-	-
2	2.0	152	2.0	-4	-2.0
3	4.0	148	2.0	-4	-2.0
4	6.0	143	2.0	-5	-2.5
5	8.0	138	2.0	-5	-2.5
6	10.0	132	2.0	-6	-3.0
7	12.0	125	2.0	-7	-3.5
8	14.0	117	2.0	-8	-4.0
9	16.0	108	2.0	-9	-4.5
10	18.0	97	2.0	-11	-5.5
11	20.0	84	2.0	-13	-6.5
12	22.0	68	2.0	-16	-8.0
13	23.0	56	1.0	-12	-12.0
14	24.0	38	1.0	-18	-18.0
15	24.5	20	0.5	-18	-36.0
16	25.0	-18	0.5	-38	-76.0
17	25.5	-48	0.5	-30	-60.0
18	26.0	-66	0.5	-18	-36.0
19	26.5	-76	0.5	-10	-20.0
20	27.0	-82	0.5	-6	-12.0
21	28.0	-88	1.0	-6	-6.0

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Procedure

- 1) Pipette out 25.0ml of acetic acid solution into a clean beaker
- 2) Add a pinch of quinhydrone to the solution
- 3) Connect the electrodes to the potentiometer properly
- 4) Fill the burette with standard sodium hydroxide solution
- 5) Record the initial EMF of the solution.

Result

The potentiometric titration of acetic acid against sodium hydroxide was successfully performed

Volume of NaOH required at equivalence point = 25.0 cm³

Molarity of NaOH solution = 0.1 mol dm⁻³

Strength of NaOH solution = 4.0 g dm⁻³

Aim :- To estimate the strength and amount of sodium carbonate (Na_2CO_3) present in the given solution by titrating it against a standardized hydrochloric acid (HCl) solution.

Principle :-

This experiment is based on a neutralization titration where a weak base (sodium carbonate) is titrated against a strong acid (hydrochloric acid). The neutralization occurs in two distinct, consecutive steps.

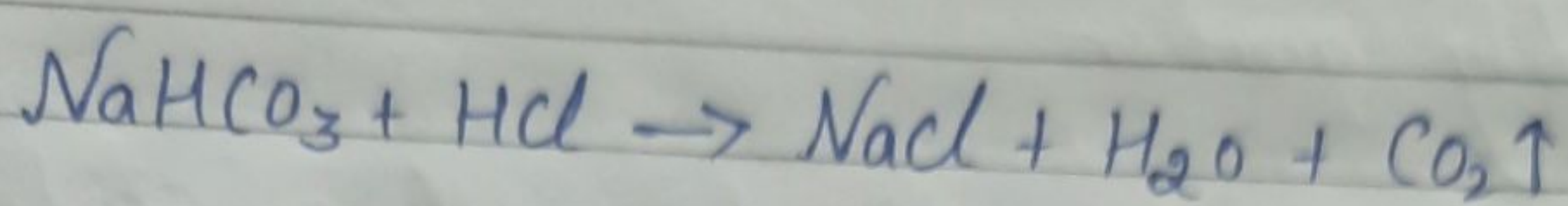
Step 1

Sodium carbonate reacts with HCl to form sodium bicarbonate (NaHCO_3). This step is completed at a weakly alkaline pH of around 8.3.

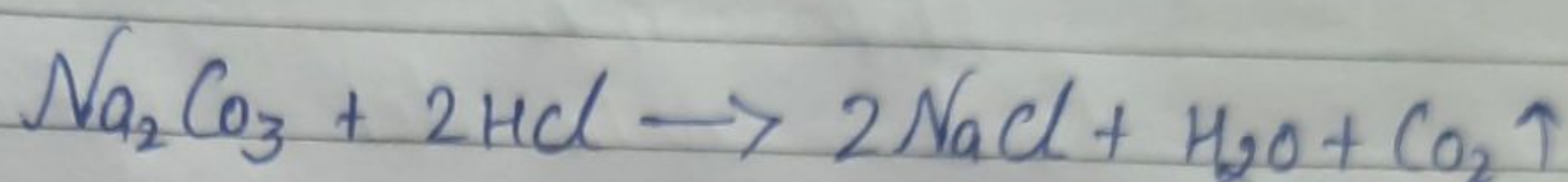


Step 2

The formed sodium bicarbonate reacts further with excess HCl to produce sodium chloride, water, and liberate carbon dioxide gas. This step goes to completion at an acidic pH of around 3.8.



Overall Balanced Equation



Choice of indicators

Because this reaction occurs in two stages, two different indicators can be utilized depending on the endpoint you wish to track.

Phenolphthalein

It changes colour from pink to colorless at a pH range of 8.0 - 10.0. This corresponds exactly to the completion of step 1 (half-neutralization)

Methyl orange

It changes color from yellow to reddish-pink at a pH range of 3.1 - 4.4. This marks the completion of step 2 (Complete neutralization)

In a standard total-estimation experiment, Methyl orange is chosen as the primary indicator because it ensures that both equivalents of the carbonate ion are fully neutralized.

Requirements

Apparatus

- Burette (50cm^3)
- Volumetric flask ($100\text{cm}^3/250\text{cm}^3$)
- Pipette (20.00 or 25.00cm^3)
- Burette stand and clamp
- Conical flasks (250cm^3)
- Funnel and glass rod

Chemicals

- Standardized hydrochloric acid solution (HCl , approx. 0.1M)
- Unknown sodium carbonate (Na_2CO_3) solution
- Methyl orange indicator
- Distilled water

Procedure

- 1) Wash all glass apparatus thoroughly with distilled

Calculation

1) Normality Equation

According to the law of chemical equivalence

$$N_1 V_1 = N_2 V_2$$

(Sodium Carbonate) = (Hydrochloric acid)

Where, N_1 = Normality of unknown Na_2CO_3 solution

V_1 = Volume of Na_2CO_3 solution = 25.0 cm^3

N_2 = Normality of standardized HCl solⁿ = 0.1 N

V_2 = Volume of HCl used = 24.5 cm^3

2) Calculation of Normality of Na_2CO_3

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

$$N_1 = (0.1 \times 24.5) / 25.0$$

$$N_1 = 0.098 \text{ N}$$

3) Calculation of Strength of Na_2CO_3

$$\text{Strength (g/L)} = \text{Normality} \times \text{Equivalent weight}$$

$$\begin{aligned} \text{Equivalent weight of } \text{Na}_2\text{CO}_3 &= \text{Molecular weight} / \text{valency factor} \\ &= 106 / 2 = 53 \text{ g/e} \end{aligned}$$

Therefore

$$\text{Strength} = 0.098 \times 53 = 5.194 \text{ g/L}$$

water. Rinse the burette with the standardized HCl solution.

- 2) Pipette out exactly 25.0 cm^3 of the unknown sodium carbonate solution into a clean, rinsed conical flask.
- 3) Add 2-3 drops of Methyl orange indicator to the flask.
- 4) The solution will turn a distinct yellow color.
- 5) Note down the final burette reading.

Observation Table

Volume of Na_2CO_3 solution taken (V_1) = 25.0 cm^3

S.No	Volume of Na_2CO_3 (V_1 , cm^3)	Initial burette Reading (cm^3)	Final burette Reading (cm^3)	Volume of HCl Added (V_2 , cm^3)	Concordant Reading (cm^3)
1	25.0	0.0	24.6	24.6	24.5
2	25.0	0.0	24.5	24.5	
3	25.0	0.0	24.5	24.5	

Concordant volume of standardized HCl used (V_2) = 24.5 mL

Result

The normality of the given sodium carbonate solution was found to be 0.098 N

The strength of sodium carbonate solution was found to be 5.194 g/dm³

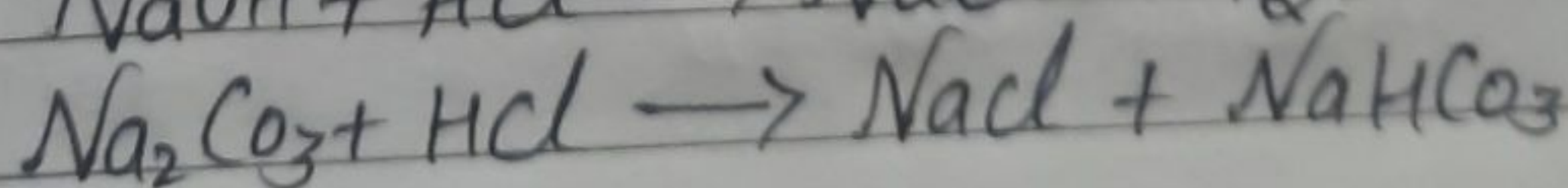
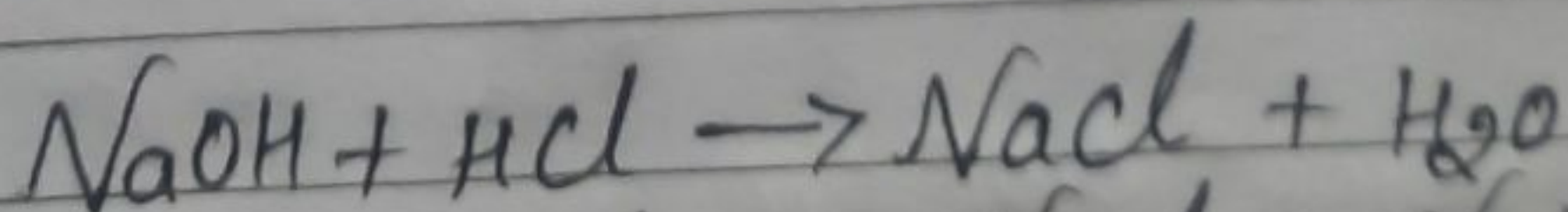
Aim - To determine volumetrically the amount of sodium carbonate (Na_2CO_3) and sodium hydroxide (NaOH) present together in the given alkali mixture solution using a standard 0.1 NHCl solution

Theory :-

This analysis is based on a two-stage sequential neutralization process utilizing two distinct indicators that react at different pH thresholds.

Stage 1 :- Titration using phenolphthalein indicator

When the alkali mixture containing phenolphthalein indicator is titrated against standard 0.1 N-HCl , the bright pink colour of the indicator gets discharged (turns colourless) at a pH of around 8.3. This occurs exactly when the whole of sodium hydroxide (NaOH) and one-half of sodium carbonate (Na_2CO_3) present in the solution get neutralized.

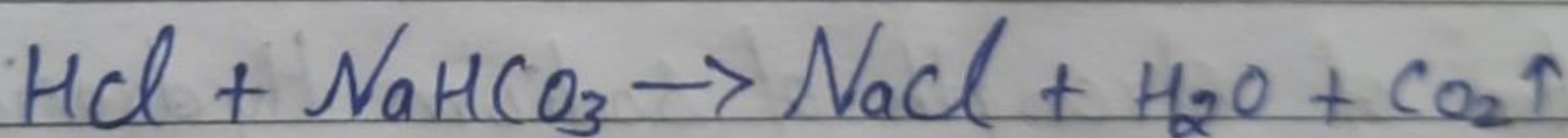


Let the volume of 0.1 N HCl required to reach this stage be V_1 mL.

Stage 2 :- Titration using Methyl orange indicator.

To the above neutralized colorless solution, 2-3 drops of methyl orange are added, turning the solution yellow. Further quantity of hydrochloric acid is run down from the burette until a pink (or orange) color is obtained at a pH of around 3.8.

This additional quantity of acid corresponds strictly to the amount required to neutralize the remaining half of the carbonate.



Let this additional volume of 0.1 N HCl added in the second stage be V_2 mL.

Procedure

- 1) Rinse and fill a clean burette with standard 0.1 N HCl solution up to the zero mark.

Calculation

a) Calculation of strength of sodium carbonate (Na_2CO_3)
Total volume of acid required for complete neutralization of Na_2CO_3 present in 20 mL alkali mixture = $2V_2 = 2 \times 5.0 = 10.0 \text{ mL}$

using normality relation :-

$$20 \times N_{\text{Na}_2\text{CO}_3} = 10.0 \times 0.1$$

Therefore

$$N_{\text{Na}_2\text{CO}_3} = (10.0 \times 0.1) / 20$$

$$N_{\text{Na}_2\text{CO}_3} = 0.05 \text{ N}$$

Strength of Na_2CO_3

$$\text{Strength} = \text{Normality} \times \text{Equivalent weight}$$

$$\text{Equivalent weight of } \text{Na}_2\text{CO}_3 = 53 \text{ g/equivalent}$$

Therefore

$$\text{Strength} = 0.05 \times 53 = 2.65 \text{ g/litre}$$

b) Calculation of strength of sodium Hydroxide (NaOH)
Volume of acid required for complete neutralization of NaOH present in 20 mL alkali mixture

$$= (V_1 - V_2) = 18.4 - 5.0 = 13.4 \text{ mL}$$

using normality relation : $20 \times N_{\text{NaOH}} = 13.4 \times 0.1$

Therefore,

$$N_{\text{NaOH}} = (13.4 \times 0.1) / 20$$

$$N_{\text{NaOH}} = 0.067 \text{ N}$$

- 2) Pipette out exactly 20ml of the given alkali mixture solution into a clean 250ml conical flask
- 3) Add 2-3 drops of phenolphthalein indicator to the flask.
- 4) The solution immediately turns pink.

Observation Table

Volume of alkali mixture solution pipetted out (V_{mix}) = 20ml

Normality of standard hydrochloric acid (N_{HCl}) = 0.1N

Titration of Alkali Mixture against 0.1N HCl

S.No	Volume of mixture (mL)	Phenolphthalein Endpoint Reading V_1 (mL)	Methyl orange Final Reading ($V_1 + V_2$) (mL)	Additional Methyl orange volume V_2 (mL)
1	20.0	18.5	23.6	5.1
2	20.0	18.4	23.4	5.0
3	20.0	18.4	23.4	5.0

Concordant volume values

Strength of NaOH

Strength = Normality $\times$ Equivalent weight
Equivalent weight of NaOH = 40 g/equivalent

Therefore,

$$\text{Strength} = 0.067 \times 40 = 2.68 \text{ g/litre}$$

Normality	Equivalent weight	Strength (g/litre)
0.067	40	2.68
0.067	40	2.68
0.067	40	2.68

V_1 (Volume up to phenolphthalein endpoint) = 18.4 mL
 V_2 (Additional volume for methyl orange endpoint) = 5.0 mL

Result :-

In the given unknown alkali mixture solution
Strength of Sodium carbonate (Na_2CO_3) = 2.65 g/lit.
Strength of sodium hydroxide (NaOH) = 2.68 g/lit.

Aim :- To determine the concentration and total amount of sodium (Na^+) ions present in the given unknown sample solution flame photometrically using the calibration curve method.

Principle :-

Flame photometry (also known as flame emission spectroscopy) is an analytical technique concerned with the emission of characteristic radiation in flames by individual elements and correlating the emission intensity with the concentration of these elements.

Desolvation -

Evaporation of the solvent leaving behind a fine mist of solid solute particles.

Vaporization :-

Vaporization of the solute particles into gaseous molecules.

Atomization :-

Dissociation of the solute compounds to form free ground-state atoms (Na^0)

Excitation :-

Thermal energy of the flame excites the ground-state atoms to unstable higher energy levels.

Emission :-

The unstable excited atoms quickly return to lower energy states by emitting photons of characteristic wavelength, producing a yellow flame.

The intensity of emitted light is directly $\propto$ to the concentration of sodium ions present in the solution.

$$I \propto C$$

The emitted light passes through a sodium-specific optical filter that allows only wavelength of 589 nm to reach the detector.

Requirements

Apparatus

- flame photometer
- Air compressor
- Fuel gas source
- Volumetric flasks (100 mL and 12)

- Pipettes
- Beakers
- Distilled water

Chemicals & Reagents

- i) Stock sodium ion solution (100 ppm)

Prepared by dissolving 0.2542 g of AR-grade sodium chloride (NaCl) in distilled water and making the volume up to 1 litre.

- ii) unknown sample solution containing sodium ions.

Experimental working conditions

- Gas mixture used: Air + Acetylene
- Air pressure maintained at 10 lbs/sq inch
- Sodium filter wavelength = 589 nm

5 Procedure

I Preparation of working standards

- Take five clean 100 mL volumetric flasks labeled 1 to 5
- Make up the volume in each flask to 100 mL with

Calculation

1) Concentration of Na^+ ions

$$X = 13.0 \text{ ppm}$$

$$= 13.0 \text{ mg/L}$$

2) Total amount of Na^+ ions present

Amount of Na^+ ions (mg)

$$= (\text{concentration} \times \text{volume}) / 1000$$

$$= (13.0 \times 100) / 1000 \Rightarrow 1.3 \text{ mg}$$

Converting into grams :-

Amount of Na^+ ions

$$= 1.3 / 1000 \Rightarrow 0.0013 \text{ g}$$

distilled water.

II Instrument Calibration and Measurement

- Switch on the flame photometer and allow it to warm for about 10 minutes.
- Ignite the burner and adjust the air-fuel ratio properly
- Insert the sodium filter of wavelength 589nm.
- Aspirate distilled water and adjust the zero reading.

Observation Table

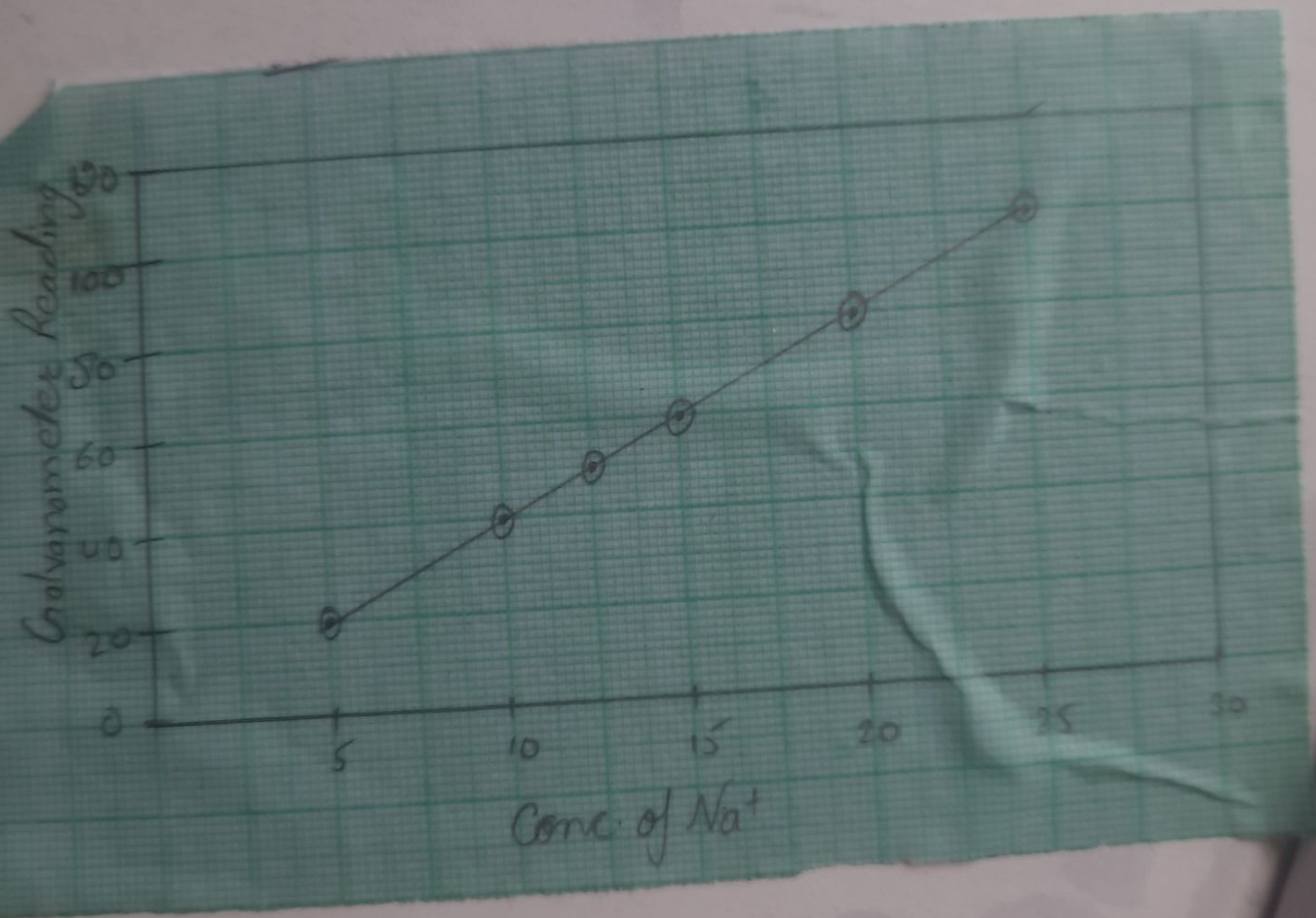
Volume of unknown sample diluted up to (V_{final}) = 100 mL

Operational wavelength for sodium (λ_{Na}) = 589 nm

Flame Photometric intensity Readings

Flask No.	Volume of stock Solution Add (mL)	Total volume (mL)	Concentration (ppm)	Galvanometer Reading
1	5.0	100	5	20
2	10.0	100	10	41
3	15.0	100	15	60

Teacher's Signature _____



4	20.0	100	20	81
5	25.0	100	25	100
6	Exactly 100 ml	100	λ	52

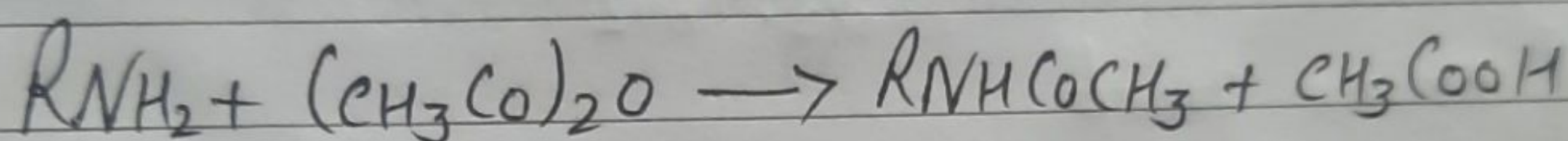
Result:-

The concentration of sodium (Na^+) ions in the given unknown sample solution determined by flame photometry is 13.0 ppm (13.0 mg dm^{-3})

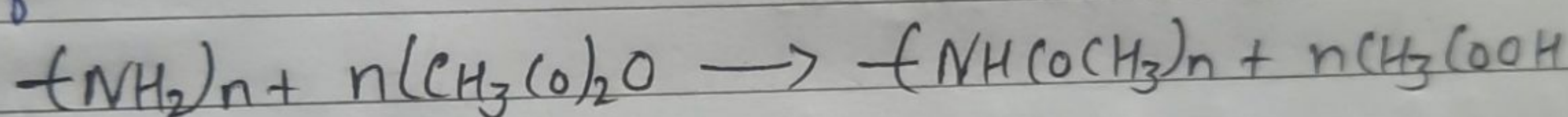
The total amount of sodium ions present in the given sample solution is 0.0013g (1.3mg)

Aim :- To estimate the percentage of amino groups present in the given sample of an unknown amino compound (Aniline) by back-titration using the acetylation method.

Theory :- Primary and secondary amino compounds react quantitatively with a known excess of an acetylating agent to form an acetyl derivative and free acetic acid.



or



The reaction mixture is heated with an excess of the acetylating mixture to ensure the conversion is complete.

Reagents

i) Acetylating Mixture :- Prepared by thoroughly mixing one volume of pure acetic anhydride with three volumes of dry pyridine

ii) Standard $N/2$ (0.5N) Alcoholic NaOH solution :-
Standardized titrant solution

iii) Indicator :- Phenolphthalein solution

Procedure

- 1) Weigh a clean, dry 100 mL round-bottom flask accurately.
- 2) Pipette exactly 10.0 mL of the prepared acetylating mixture into the flask.
- 3) Remove the flask from the water bath and allow it to cool slightly.
- 4) Record the volume consumed at the permanent light pink endpoint as V_1 mL.
- 5) Record this blank volume consumed as V_2 mL.

Observation Tables :-

Calculation

i, Calculation of percentage of Amino groups.

$$\% \text{ of amino group in sample} = [(V_2 - V_1) / (10 \times w)] \times (N_H)$$

Given

$$V_2 = 48.9 \text{ mL}$$

$$V_1 = 23.2 \text{ mL}$$

$$w = 1.20 \text{ g}$$

$$\text{Normality of NaOH} = 0.5 \text{ N}$$

$$V_2 - V_1 = 48.9 - 23.2$$

$$= 25.7 \text{ mL}$$

% of amino group

$$= [25.7 / (10 \times 1.20)] \times 0.5 =$$

ii, Calculation of Number of amino groups per molecule

Molecular weight of aniline ($C_6H_5NH_2$) = 93.13 g/mol

Number of amino groups

$$= [(V_2 - V_1) \times M / (1000 \times w)] \times (\text{Normality of NaOH})$$

$$= [(25.7 \times 93.13) / (1000 \times 1.20)] \times 0.5 = (2393.44 / 1200) \times 0.5$$

$$= 1.9945 \times 0.5 = 0.997 \approx 1$$

Table 1 :- Sample Titration (With Amine)

Mass of unknown amine sample taken (w) = 1.20g

Normality of standard NaOH solution used = 0.5N

Trial No.	Mass of Sample (w, g)	Initial Burette Reading (mL)	Final burette Reading (mL)	Volume of NaOH used (V ₁ , mL)	Concordant value (V ₁ , mL)
1	1.20	0.0	23.4	23.4	
2	1.20	0.0	23.2	23.2	23.2
3	1.20	0.0	23.2	23.2	

Table 2 : Blank Titration (Without Amine)

Trial No.	Mass of Sample (w, g)	Initial Burette Reading (mL)	Final Burette Reading (mL)	Volume of NaOH used (V ₁ , mL)	Concordant value (V ₁ , mL)
1	1.20 10.0	0.0	23.4 49.1	23.4 49.1	
2	1.20 10.0	0.0	23.2 48.9	23.2 48.9	23.2 48.9
3	1.20 10.0	0.0	23.2 48.9	23.2 48.9	

Concordant volume for sample titration (V₁) = 23.2 mLConcordant volume for blank titration (V₂) = 48.9 mL

Result:-

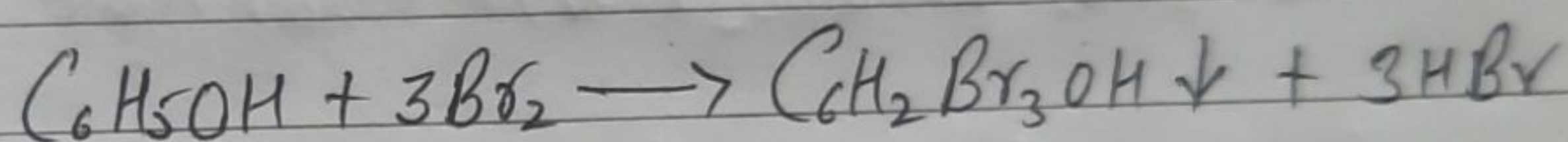
The total percentage of amino ($-NH_2$) group present in the given unknown organic ~~group~~ amino sample is 17.14%.

The calculated number of amino groups per molecule of the compound is 1.

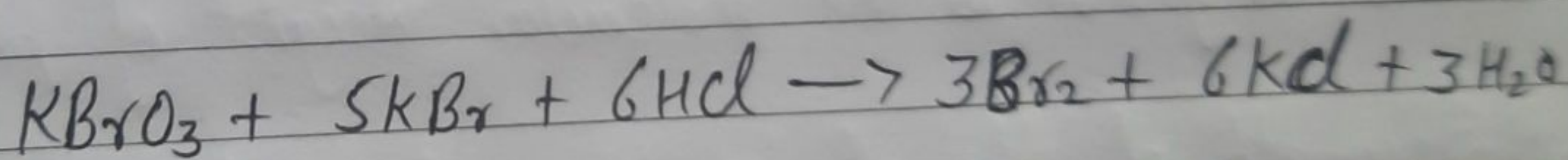
Aim :- To determine the strength and amount of phenol present in grams per litre (g/litre) in the given unknown solution using a standard bromate-bromide solution.

Theory :-

Phenols possessing free ortho- & para- position react quantitatively with an excess of a potassium bromate-potassium bromide mixture in an acidic medium.



The generation of bromine from the bromating mixture under acidic conditions takes place as follows



The total unreacted excess bromine left over in the reaction mixture is estimated using an indirect iodometric back-titration.

Reagents

- i) Brominating Mixture ($\sim 0.1N$):- Prepared by dissolving $0.4g$ potassium bromate and $1.8g$ potassium bromide in distilled water and making the volume up to $100ml$.
- ii) Sodium Thiosulphate solution - Prepared by dissolving $6.2g$ sodium thiosulphate in distilled water and making the volume up to $250ml$.
- iii) Potassium Iodide solution (20%):- Prepared by dissolving $15g$ KI in $75ml$ distilled water.
- iv) Indicator:- Freshly prepared 1% starch solution.

Procedure

- 1) Add exactly $25ml$ brominating mixture followed by $25ml$ distilled water.
- 2) Add 2-3 drops of starch indicator.

Calculation

1000ml of 1N Hypo solution = 1g equivalent weight of hypo
= 1g equivalent weight of bromine

Therefore, the volume difference $(V_2 - V_1)$ ml of N hypo solution is given by:

Equivalent weight of Br_2
 $= [(V_2 - V_1) / 1000] \times (N)$ g equivalent weight of bromine

Since 1 mole of phenol requires 3 moles of Br_2

Moles of phenol neutralized

$$= [(V_2 - V_1) / 1000] \times (N) \times (1/6) \text{ mole of phenol}$$

1) Mass of phenol present in 25ml sample

Mass of phenol in 25ml

$$= [(V_2 - V_1) / 1000] \times (N) \times (94/6) \text{ g}$$

where 94 g/mol is the molecular weight of phenol, $\text{C}_6\text{H}_5\text{OH}$

2) Strength of phenol per litre of solution

$$\text{Amount of phenol (g/litre)} = [(V_2 - V_1) / 1000] \times (N/x) \times (94/6) \times (1000/25)$$

Simplifying

$$\text{Amount of phenol (g/litre)}$$

$$= [(V_2 - V_1) / 150] \times (N) \times 94$$

3) The solution turns deep blue.

Observation Tables

Table 1 :- Sample Titration (with phenol solution)

Volume of diluted phenol sample titrated = 25 mL

Normality of sodium thiosulphate solution (N) = 0.1N

Trial No.	Volume of Phenol Sample (mL)	Initial Reading (mL)	Final Reading (mL)	Volume of Hypo used V_1 (mL)	Concordant value (mL)
1	25.0	0.0	11.6	11.6	
2	25.0	0.0	11.5	11.5	11.5
3	25.0	0.0	11.5	11.5	

Table 2 :- Blank Titration (without phenol solution)

Trial No.	Volume of Distilled water (mL)	Initial Reading (mL)	Final Reading (mL)	Volume of Hypo used V_2 (mL)	Concordant value (mL)
1	25.0	0.0	35.6	35.6	
2	25.0	0.0	35.5	35.5	35.5
3	25.0	0.0	35.5	35.5	

Substituting Experimental Reading

$$V_2 = 35.5 \text{ mL}$$

$$V_1 = 11.5 \text{ mL}$$

$$N = 0.1 \text{ N}$$

$$V_2 - V_1 = 35.5 - 11.5 = 24.0 \text{ mL}$$

Amount of phenol (g/litre)

$$= (24.0/150) \times 0.1 \times 94 = 0.16 \times 0.1 \times 94$$

$$= 0.016 \times 94$$

$$= 1.504 \text{ g/litre}$$

Volume of sample (mL)	Initial Reading (mL)	Final Reading (mL)	Volume of hypo used (mL)
25.0	0.0	16.5	16.5
25.0	0.0	16.2	16.2
25.0	0.0	16.2	16.2

Table 2: Blank Titration (Without phenol solution)

Volume of sample (mL)	Initial Reading (mL)	Final Reading (mL)	Volume of hypo used (mL)
25.0	0.0	22.8	22.8
25.0	0.0	22.2	22.2
25.0	0.0	22.2	22.2

Concordant volume for sample titration (V_1) = 11.5 ml

Concordant volume for blank titration (V_2) = 35.5 ml

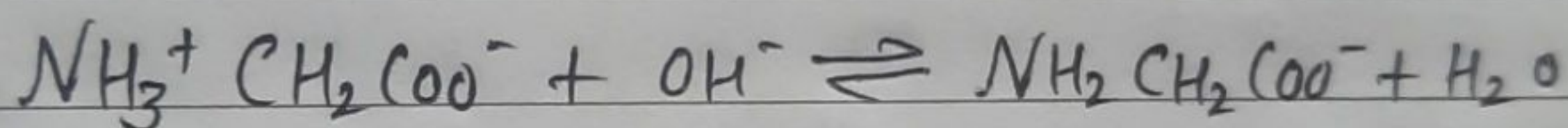
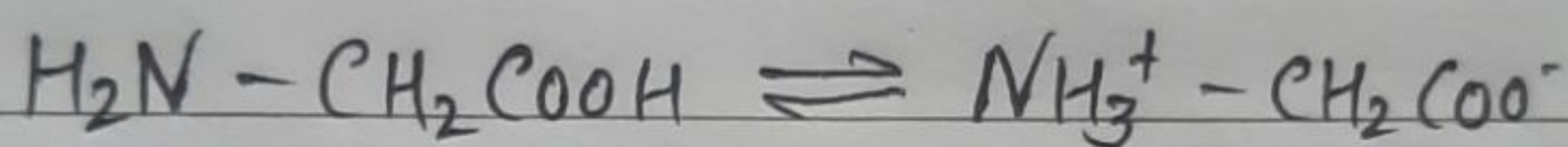
Result

The total strength of phenol present in the given unknown solution determined via the bromate-bromide iodometric back-titration method is 1.504 g/litre

Aim :- Determination of the concentration of glycine by Sorenson's formalin method

Principle :-

Amino acids like glycine, alanine, etc., contain one basic amino group ($-NH_2$) and one acidic carboxylic group ($-COOH$) as part of their structure. These groups, being of opposite nature, neutralise one another intramolecularly and form internal cyclic salts called zwitterions or dipolar ions.



Because the basic amino group interferes with the direct titration of the carboxylic acid group against a standard base, a sim

3 Requirements

Apparatus

- Burette (50cm^3)
- Pipette (25cm^3)

Teacher's Signature _____

- Volumetric flask (250cm^3)
- weighing bottle
- Wash bottle for distilled water
- Test tube
- Conical flask (250cm^3)
- funnel (small)
- Burette stand

Chemicals

- Glycine
- Formalin solution
- Sodium hydroxide
- Phenolphthalein indicator

Procedure

- 1) Weigh accurately 2 g of glycine and transfer it to a 250cm^3 Volumetric flask.
- 2) Dissolve it in distilled water and make up to the graduation mark.
- 3) Add 3-4 drops of phenolphthalein indicator.
- 4) Fill the burette with the provided sodium hydroxide solution (0.1 M).
- 5) Titrate drop by drop until a very faint pink color is

just obtained

Observation

Mass of the weighing bottle

$$m_1 = 15.2420\text{g}$$

Mass of the bottle + glycine

$$m_2 = 17.2440\text{g}, m_3 = 15.2420\text{g}$$

Mass of glycine transferred

$$m = m_2 - m_3$$
$$= 17.2440 - 15.2420$$

$$= 2.0020\text{g}$$

Observation Table I

Standard Glycine Solution Vs. Sodium hydroxide Solution

Calculation

The volume of sodium hydroxide solution used for 25 cm³ of standard glycine solution

$$V_1 = 26.5 \text{ cm}^3$$

The volume of sodium hydroxide solution used for 25 cm³ of unknown glycine solution

$$V_2 = 19.8 \text{ cm}^3$$

Mass of standard glycine transferred (m) = 2.002 g

i, Amount of glycine in the given solution

Amount of glycine

$$= (m \times V_2) / V_1$$

$$= (2.0020 \times 19.8) / 26.5$$

$$= 39.6396 / 26.5$$

$$= 1.4958 \text{ g}$$

ii, Strength of the unknown glycine solution

$$\text{Strength} = (4 \times m \times V_2) / V_1 \text{ g dm}^{-3}$$

$$= (4 \times 2.0020 \times 19.8) / 26.5$$

$$= 158.5584 / 26.5$$

$$= 5.9833 \text{ g dm}^{-3}$$

Sl. No.	Volume of glycine sol ⁿ (cm ³)	Initial burette Reading (cm ³)	Final burette Reading (cm ³)	Volume of NaOH Sol ⁿ (cm ³) (Final-Initial)
1	25	0.0	26.6	26.6
2	25	0.0	26.5	26.5
3	25	0.0	26.5	26.5 (v ₁)

Observation Table II

unknown glycine solution Vs. Sodium hydroxide solution

Sl. No.	Vol ^m of unknown Glycine sol ⁿ (cm ³)	Initial burette Reading (cm ³)	Final burette Reading (cm ³)	Volume of NaOH Sol ⁿ (cm ³)
1	25	0.0	19.9	19.9
2	25	0.0	19.8	19.8
3	25	0.0	19.8	19.8 (v ₂)

Result :-

The total amount of glycine present in the given unknown sample solution is 1.4958 g.