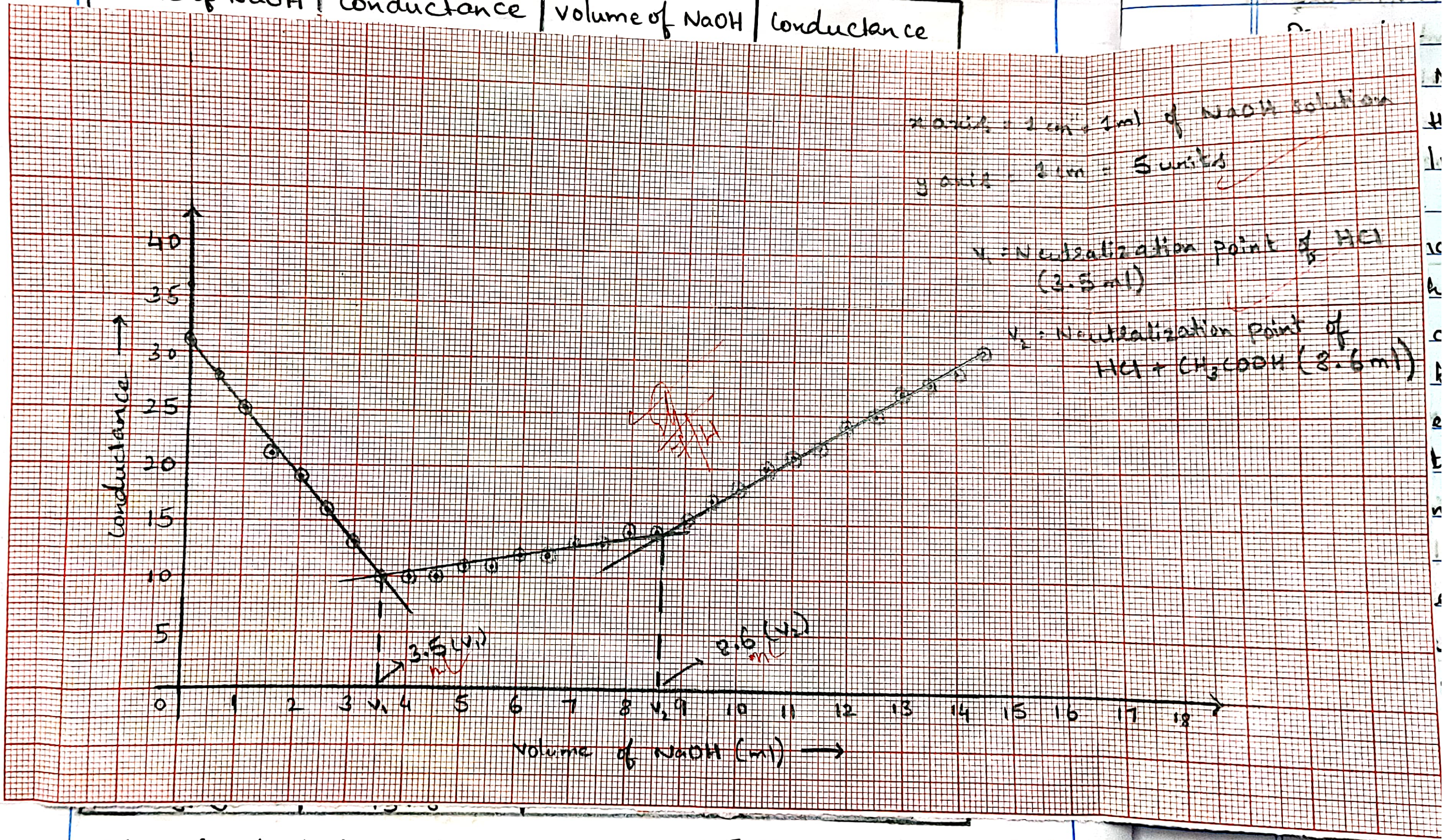
Observation

Conductometric titration of acid mixture Vs standard NaOH solution:

Volume of NaOH	Conductance	Volume of NaOH	Conductance



Note : Conductivity = conductance x cell constant [cell constant is taken as = 1]

∴ Conductivity = conductance

Experiment-1

of NaOH
(CH_3COOH

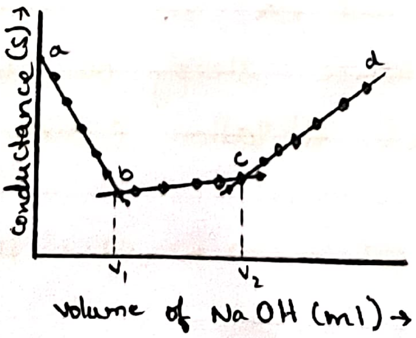
Observation

conductometric titration of acid mixture Vs standard NaOH solution:

Volume of NaOH (ml)	Conductance (S)	Volume of NaOH (ml)	Conductance (S)
0.0	30.5	8.5	14.3
0.5	27.7	9.0	15.1
1.0	24.5	9.5	16.6
1.5	21.3	10.0	18
2.0	18.5	10.5	19.6
2.5	15.7	11.0	21
3.0	13.0	11.5	22.4
3.5	10.4	12.0	24
4.0	9.6	12.5	25.3
4.5	10.2	13.0	26.7
5.0	10.7	13.5	28
5.5	11.3	14.0	29.2
6.0	11.8	14.5	30.5
6.5	12.2		
7.0	12.8		
7.5	13.3		
8.0	13.8		

Note: Conductivity = conductance x cell constant [cell constant is taken as = 1]

∴ Conductivity = conductance



V_1 = Neutralization point for HCl

V_2 = Neutralization point for HCl + CH_3COOH

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Experiment-1 Conductometric estimation of acid mixture

Principle :-

Measurement of conductance can be employed to determine the neutralization point (end point) of acid-base titration. In conductometric titration, the conductance is measured during titration and there is sudden change in conductance of the solution near the neutralization point. However, the change is not sharp and hence the neutralization point is determined graphically by plotting conductivity against volume of base. The principle underlying conductometric titrations is the replacement of ions of a particular conductivity by ions of different conductivity during titration.

Conductometric titration may be applied for the determination of amount of acids present in an acid mixture. In the titration of mixture of strong acid (HCl) and a weak acid (CH_3COOH) against strong base (NaOH), the conductivity initially decreases upon adding NaOH to acid mixture owing to the substitution of highly mobile H^+ ion of HCl by less mobile Na^+ ions of NaOH. This trend continues till all the H^+ ions of HCl are replaced by Na^+ ions (i.e strong acid, HCl is neutralized). Continued addition of NaOH raises the conductance moderately, as the weak acid (CH_3COOH) is converted into its salt (CH_3COONa) and it undergoes dissociation to give CH_3COO and Na^+ ions. Further addition of NaOH raises the conductance steeply due to the presence of free OH^- ions. A typical titration curve is shown in the figure.

$$HCl + NaOH \longrightarrow NaCl + H_2O$$

$$CH_3COOH + NaOH \longrightarrow CH_3COONa + H_2O$$

Calculations:

Strength of standard NaOH solution N_{NaOH} (given) = 1 N

Volume of acid mixture pipetted out = 25 ml

Volume of NaOH required to neutralize HCl (V_1) = 3.5 ml

Volume of NaOH required to neutralize both HCl and CH_3COOH (V_2) = 8.6 ml

Volume of NaOH required to neutralize CH_3COOH ($V_2 - V_1$) = 5.1 ml

Therefore, the strength of HCl, $N_{HCl} = \frac{1 (N_{NaOH}) \times 3.5 \; (V_1)}{25}$

$= 0.14$ N

Weight of HCl present in $1 dm^3$ of acid mixture = $N_{HCl} \times$ Eq. wt. of HCl

$= 0.14 \times 36.5 \; g/dm^3$

$= 5.11 \; g/dm^3$

Strength of CH_3COOH, $N_{Acetic\ acid} = \frac{1 (N_{NaOH}) \times 5.1 \; (V_2 - V_1)}{25}$

$= 0.204$ N

Weight of CH_3COOH present in $1 dm^3$ of acid mixture

$= N_{Acetic\ acid} \times$ Eq. wt of CH_3COOH

$= 0.204 \times 60 \; g/dm^3$

$= 12.24 \; g/dm$

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Procedure :

Pipette out 25 ml of given acid mixture into a beaker. Add one test tube distilled water. Place the conductivity cell in the acid mixture solution and connect the cell to the conductivity bridge. Insert a magnetic bead into the acid mixture Keep it on the magnetic stirrer and switch on. Note down the initial conductance value of the solution. Fill the burette with standard NaOH solution. Add NaOH from the burette in an increment of 0.5 ml and record the conductance after each addition. The conductance initially decreases and then rises slowly and finally rises sharply. Continue the addition of NaOH until the conductance is almost the same as the initial value.

Plot a graph of conductivity on y-axis against volume of NaOH added on x-axis. In figure, point 'b' corresponds to neutralization of HCl i.e. the volume of NaOH required to neutralize HCl (V_1). The point 'c' corresponds to neutralization of both HCl and CH_3COOH (V_2). The difference in the volume ($V_2 - V_1$) corresponding to the (points 'c' and 'b' volume of NaOH required to neutralize CH_3COOH.

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Result :

(1) Strength of HCl = 0.14 N

(2) weight of HCl present in 1 dm^3 of acid mixture = 5.11 g/dm^3

(3) Strength of CH_3COOH = 0.204 N

(4) Weight of CH_3COOH present in 1 dm^3 of acid mixture = 12.24 g/dm^3

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Result :

1 Strength of HCl $= \underline{0.14}$ N

2 Weight of HCl present in 1 dm^3 of acid mixture $= \underline{5.11}$ g/dm^3

3 Strength of CH_3COOH $= \underline{0.204}$ N

4 Weight of CH_3COOH present in 1 dm^3 of acid mixture $= \underline{12.24}$ g/dm^3.

30/30

4/4

Observation & Calculations:

Part A: Preparation of standard di-sodium salt of EDTA solution

Weight of weighing bottle + di-sodium salt of EDTA (m_1) = 6.3469 g

Weight of empty weighing bottle (m_2) = 3.6811 g

Weight of di-sodium salt of EDTA crystal transferred ($m_1 - m_2$) = 2.6658 g

Molarity of di-sodium salt of EDTA solution = $\frac{2.6658\ (m_1 - m_2) \times 4}{\text{Mol. wt. of di-sodium EDTA (372)}}$

= 0.0286 M (A)

Part B: Determination of total hardness of water sample

Burette : Standard di-sodium salt of EDTA solution

Conical flask : 25 ml of water sample + 3 ml buffer solution ($NH_4Cl + NH_4OH$)

Indicator : Eriochrome Black-T

Color change : Wine red to clear blue

Burette reading (ml)	Trial 1	Trial 2	Trial 3
Final burette Reading	16.3	16.4	16.4
Initial burette Reading	0.0	0.0	0.0
Volume of di-sodium salt of EDTA rundown (B)	16.3	16.4	16.4

1000 ml of 1M disodium salt of EDTA = 100 g of $CaCO_3$

(B) ml of (A) M di-sodium salt of EDTA = [16.4 (B) × 0.0286 (A) × 0.1] g of $CaCO_3$

= 0.046904 g $CaCO_3$

25 ml of water sample contains = 0.046904 g of $CaCO_3$ (C)

10^6 ml of water sample contains = $\frac{0.046904\ (C) \times 10^6}{25}$

= 1876.16 ppm of $CaCO_3$

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Experiment - 2 Estimation of Total hardness of water by EDTA method

Principle:

The hardness of water is due to the presence of dissolved salts of calcium and magnesium. Hard water does not produce much lather with soap, because Ca^{2+} and Mg^{2+} ions of hard water react with soap to form precipitate. The total hardness due to Ca and Mg salts is determined by titrating a known volume of the water sample with a standard solution of di-sodium salt of EDTA, (ethylene diamine tetra-acetic acid) using Eriochrome Black-T indicator, at a pH of ~10 (using a buffer solution of $NH_4OH - NH_4Cl$). The reaction between the metal ion ($M^{2+} = Ca^{2+}$ and Mg^{2+}) and the di-sodium salt of EDTA at pH of 10 maybe represented as:

$$M^{2+} + H_2Y^{2-} \longrightarrow MY^{2-} + 2H^+$$

The H^+ ions released, will decrease the pH of the solution. However, the coordination complex between the metal ions and EDTA is stable only at alkaline pH and therefore, a constant pH of 10 is maintained using a buffer solution of $NH_4OH - NH_4Cl$. The indicator Erichrome black-T (EBT) forms a weak wine-red complex [metal-EBT] with metal ions. The color of the free EBT indicator in the pH range 7-10 is blue.

M^{2+} + EBT $\longrightarrow$ M-EBT complex (wine red)

EBT complex + EDTA $\longrightarrow$ M-EDTA complex + Free EBT Indicator (blue)

During the titration, when EDTA is added, it reacts with the free Ca^{2+} and Mg^{2+} ions to form stable colorless Metal-EDTA complex. When the free Ca^{2+} and Mg^{2+} ions are just completely consumed, the added EDTA displaces the indicator (EBT) free from metal-EBT complex. The free indicator being blue makes the solution turn from wine red

Result: Total hardness of the given water sample = $\underline{1876.16}$ ppm of $CaCO_3$

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to blue at the end point. Knowing the volume of standard di-sodium salt of EDTA solution used up in the titration, the hardness of water is calculated in terms of 'parts of $CaCO_3$ per million of water' (ppm) [i.e. Parts of $CaCO_3$ equivalent per 10^6 parts of water].

Procedure:

Part A: Preparation of standard solution of di-sodium salt of EDTA:

Weigh out the given di-sodium salt of EDTA crystals accurately in a weighing bottle. Transfer all the crystals into a clean 250 ml volumetric flask through a funnel. Record the weight of the weighing bottle after transferring the crystals. Add 5 ml of NH_4OH solution. Wash the funnel with distilled water so that all the crystals are transferred quantitatively into the flask. Add adequate quantity (100 ml) of distilled water into the flask to dissolve the crystals completely, and dilute up to 250 ml mark and close the stopper. Mix the solution well for homogeneity. Calculate the molarity of di-sodium salt of EDTA solution.

Part B: Determination of total hardness of water sample:

Fill the burette with standard EDTA solution. Pipette out exactly 25 ml of the given water sample into a clean conical flask. Add 3 ml of $NH_4Cl - NH_4OH$ buffer mixture ($\sim pH = 10$) and a pinch of Eriochrome black-T indicator. Solution color changes to wine red. Carefully titrate against standard di-sodium salt of EDTA solution. At the end point, Eriochrome black-T changes color from wine red to clear blue. Note down the volume of di-sodium salt of EDTA consumed. Repeat the titration 3 times for concordant values. From the volume of EDTA consumed,

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calculate the total hardness of the given water sample.

Result: Total hardness of the given water sample = 1876.16 ppm of $CaCO_3$

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