|| JAI SRI GURUDEV ||

BGS INSTITUTE OF TECHNOLOGY

(Approved by AICTE, New Delhi and Recognized by Govt. of Karnataka)

BG Nagara - 571 448 (Bellur Cross), Nagamangala Taluk, Mandya District

Certificate

This is to certify that Mr/Ms. Indushree H.S

USN 19CSE028 has satisfactorily completed the course of experiments in Engineering chemistry Laboratory (Course Code 18CHEL16) prescribed by the Adichunchanagiri University, for I st Semester, BE Computer Science Engineering, of this College in the year 2019-2020

Record Marks : 30 Test Marks : 10

IA Marks : 40

Date : 03/12/19

Staff Incharge

Head of the Department

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Name of the Student : Indushree H.S Class : D Sem. Ist

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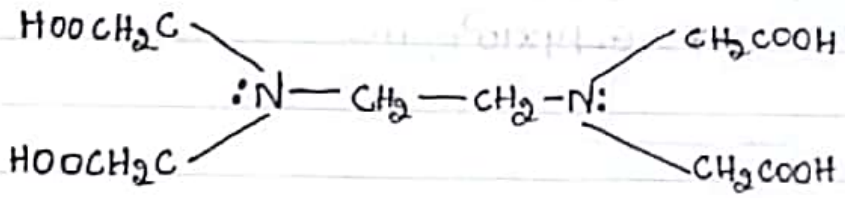
$\frac{94}{300}$ $\frac{30}{30}$

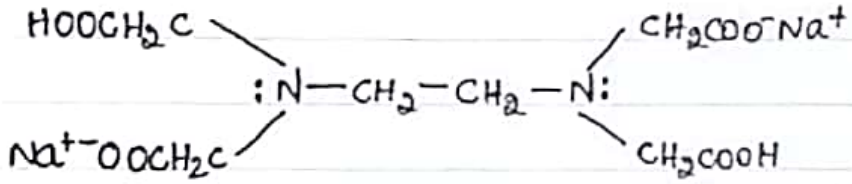
DETERMINATION OF TOTAL HARDNESS OF WATER

THEORY

Hardness of water is due to the presence of dissolved salts of calcium and magnesium. The total hardness of water is determined using ethylene diamine tetra acetic acid (EDTA) which form complex with Ca^{2+} and Mg^{2+} ions. The EDTA molecule has two easily replaceable hydrogen atoms and hence the EDTA form complex with metal ion present in water. Reaction can be carried out quantitatively at a pH of 10 using Eriochrome black-T as indicator. Since the reaction involves the liberation of H^+ ions, a buffer mixture has to be used to maintain a pH of 10. The buffer mixture used in the titration is ammonium hydroxide and ammonium chloride. The total hardness of the water is usually expressed in terms of ppm (i.e parts per million) of $CaCO_3$. EDTA is sparingly soluble in water and hence disodium salt of EDTA is used as reagent for preparing the solution. The structures of EDTA (H_4Y) and disodium salt of EDTA (Na_2H_2Y) are given below.



EDTA (H_4Y)



Disodium salt of EDTA (Na_2H_2Y)

PROCEDURE

1. Preparation of standard solution of disodium salt of EDTA

When Weigh given disodium salt of EDTA accurately using an electronic balance. Transfer the crystals of EDTA into a clean 250cm³ standard flask using a funnel. To this add one test tube of ammonium hydroxide and little distilled water and shake well to dissolve the crystals. Make up the solution to the Mark by adding distilled water and mix well for uniform concentration calculate the molarity of EDTA solution.

2. Determination of total hardness of water

Pipette out 25cm³ of the given hard water sample into a clean conical flask, to this add 5cm³ of ammonia—ammonium chloride buffer solution, and two drops of Eriochrome black-T indicator. Titrate this mixture against standard EDTA solution taken in a burette untill the wine red color changes to clear blue. Note down the volume of EDTA added and repeat the experiment for concordant values. Calculate the total hardness of water in terms of ppm.

REACTIONS

M^{2+} (Ca^{2+} or Mg^{2+}) + In (indicator) $\longrightarrow$ M-In (wine red color) (metal ion - In complex)

M - In + Na_2H_2Y $\longrightarrow$ M-Y + $2Na^+$ + $2H^+$ + In
(blue) (metal ion - EDTA complex)

RESULT:

The total hardness of the given water sample is 6.44x10³ ppm of $CaCO_3$

OBSERVATIONS AND CALCULATIONS

1. Preparation of standard solution of disodium salt of EDTA

Weight of weighing bottle + EDTA salt = 6.8689 g

Weight of empty weighing bottle = 4.6232 g

∴ Weight of EDTA salt taken = 2.2457 g

$$\text{Molarity of EDTA} = \frac{\text{Weight of EDTA salt}}{\text{Molecular weight of EDTA}} \times 4$$

$$M_{EDTA} = \frac{2.2457}{372.24} \times 4 = 0.02413 (a) M$$

2. Determination of total hardness of water.

Burette : Standard EDTA solution

Conical flask : 25 cm^3 of hard water + 5 cm^3 of $NH_4OH - NH_4Cl$ buffer.

Indicator : Eriochrome black-T

End point : Wine red to clear blue colour.

Burette readings (cm^3)	I	II	III
Final readings	7.0	14.0	21.0
Initial readings	0.0	7.0	14.0
Volume of EDTA run down	7.0	7.0	7.0

$\therefore$ volume of EDTA required $V = \underline{7.0}\ cm^3$

<u>NOTE</u>: Molecular weight of $CaCO_3 = 100$

$1000 cm^3$ of 1M of EDTA = one g molecular weight of $CaCO_3$(100)

$\therefore V\ cm^3$ of (a)M EDTA $= X = \frac{(a) \times V \times 100}{1000} =$

$$= \frac{(0.024)(7)100}{1000}$$

$$= \frac{16.8}{1000}$$

$$X = \underline{0.0168 \times 10^{-3} g} \text{ of } CaCO_3$$

$\therefore$ Weight of $CaCO_3$ present in $25 cm^3$ of hardWater =

$$X = \underline{0.0168 \times 10^{-3} g}$$

$\therefore$ Weight of $CaCO_3$ present in $25 cm^3$ of hard water in terms of ppm $= Y = \frac{X \times 10^6}{25}$

$$= \frac{0.0168 \times 10^{-3} \times 10^6}{25}$$

$$Y = 6.74 \times 10^3 ppm$$

Thus total hardness of water in terms of ppm

$Y = \underline{6.74 \times 10^3 \text{ ppm}}$

Name of Experiment

Date 03 08 19

Experiment No. 02

Page No. 04

DETERMINATION OF CALCIUM OXIDE IN THE GIVEN SAMPLE OF CEMENT SOLUTION BY RAPID EDTA METHOD

THEORY

Cement contains oxides of calcium, aluminum, magnesium, iron and small amount of silica. calcium oxide is a prime constituent of cement and the general composition of portland cement is given below

$CaO = 60-66\%$, $SiO_2 = 17-25\%$, $Al_2O_3 = 3-8\%$, $Fe_2O_3 = 2-6\%$, $MgO = 0.1-5.5\%$, $SO_3 = 1-3\%$ and Na_2O & $K_2O = 0.5-1.5\%$

In the estimation of calcium in cement, the given cement sample is treated with concentrated hydrochloric acid. the insoluble silica is filtered off and the filtrate which contains calcium ions is titrated against EDTA at a pH of 12-14 using patterson-Reeder's indicator. the latter is used in this titration because Eriochrome black-T indicator forms a very weak complex with calcium ions. Diethylamine is added to maintain a pH of about 12.5 and glycerol is added to get a sharp end point. This method facilitates the determination of calcium only although Mg^{2+} ions are present in the cement solution which can readily form complexes with EDTA.

(i.e., Mg^{++} ions do not react with EDTA in the above conditions)

PROCEDURE

1. PREPARATION OF STANDARD SOLUTION OF DISODIUM SALT OF EDTA

Weigh the given disodium salt of EDTA accurately using an electronic balance. Transfer the crystals of EDTA into a clean $250cm^3$ standard flask using a funnel. Dissolve the crystals by adding one t.t of ammonium hydroxide and little distilled water. Make up the solution of the mark by adding distilled water and mix well for uniform concentration. calculate the molarity of EDTA solution.

2. Estimation of calcium oxide

Pipette out $25cm^3$ of the given cement solution into a clean conical flask add $5cm^3$ of 1:1 glycerol, $5cm^3$ of diethyl amine and $10cm^3$ of 4N sodium hydroxide. Add 3-4 drops of pattern-Reeder's indicator and titrate this against EDTA solution taken in a burette until wine red color changes to purple blue. Note down the volume of EDTA added.

and repeat the experiment for concordant values. Calculate the percentage of calcium oxide in the given cement solution.

REACTIONS

$$\underset{\text{cement}}{(CaO + SiO_2)} + 2HCl \rightarrow CaCl_2 + SiO_2\downarrow + H_2O$$

$$Ca^{2+} + In \rightarrow Ca\text{-}In \text{ complex (wine red colour)}$$

$$Ca\text{-}In + EDTA \rightarrow Ca\text{-}EDTA \text{ complex} + In \text{ (blue colour)}$$

Result

The amount of CaO present in the given cement solution is 0.4812g

29/30 03/09/19

OBSERVATIONS AND CALCULATIONS

1. Preparation of standard solution of disodium salt of EDTA

Weight of weighing bottle + EDTA salt =

Weight of empty weighing bottle =

∴ Weight of EDTA salt taken =

$$\text{Molarity of EDTA} = \frac{\text{Weight of EDTA salt}}{\text{Molecular weight of EDTA}} \times 4$$

$$M_{EDTA} = \frac{2.1031 \times 4}{372.24} = 0.02259 (a) M$$

2. Estimation of CaO in cement solution

Burette : Standard EDTA solution

Conical flask : 25cm³ of cement solution + 5cm³ of 1:1 glycerol + 5cm³ of di-ethylamine + 10cm³ of 4N NaOH

Indicator : pattern – Reeder's indicator

END point : Wine red-to purple blue colour.

Name of Experiment

Experiment No. 02

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Burette readings	I	II	III
Final readings	9.5	9.5	9.5
Initial readings	0.0	0.0	0.0
Volume of EDTA run down in cm^3	9.5	9.5	9.5

$\therefore$ volume of EDTA required $= V = \underline{9.5\,cm^3}$

<u>Note</u>: Molecular weight of CaO = 56.08

1000 cm^3 of 1M of EDTA = One gm molecular weight of CaO (56.08)

$\therefore V cm^3$ of (a) M EDTA $= X = \dfrac{(a) \times V \times 56.08}{1000}$

$= \dfrac{0.225 \times 9.5 \times 56.08}{1000}$

$X = \underline{0.0120}\,g$ of CaO

$\therefore$ weight of CaO present in $25 cm^3$ of cement solution =

$X = 0.01203\,g$

$\therefore$ weight of CaO present in $1000 cm^3$ of cement solution

$Y = \dfrac{X \times 1000}{25}$

$Y = \dfrac{0.01203 \times 1000}{25}$

$Y = \underline{0.4812}\,g$

DETERMINATION OF PERCENTAGE OF COPPER IN BRASS USING STANDARD SODIUM THIOSULPHATE SOLUTION.

THEORY:

The chief constituents of brass alloy are copper and zinc. It also contains small quantities of tin, lead and iron. The percentage composition of a typical brass alloy is given below

Cu = 50 - 90%, Zn = 30 - 40%, Sn = 0 - 6%, Pb = 0 - 2% and Fe = 0 - 1%.

A solution of brass is made by dissolving the sample in minimum amount of nitric acid and excess of nitric acid and excess of nitric acid destroy-ed by boiling with urea. The solution is neutralize-d and made in to slight acidic medium. Potassium iodide solution is added and the cupric ion present in the brass solution oxidise potassium iodide to iodine. The liberated iodine is titrated against sodium thiosulphate using starch as indicat-or. The volume of sodium thiosulphate consumed is mesure of the amount of copper pousent in the solution and sodium thiosulphate which is commonly known as hypo.

PROCEDURE:

1. Preparation of Brass solution

Weigh the given brass pieces accurately weigh and transfer it into a clean conical flask. Add one t.t of 1:1 nitric acid. warm gently to dissolve the brass piece completely and add two t.t of distilled water. Add pinch of urea and continue the heating until the reddish brown fumes of oxides of nitrogen are expelled completely. cool the solution to room temperature and transfer the solution to the mark by adding distilled water and mix well for uniform concentration.

2. Estimation of copper:

Pipette out 25cm³ of brass solution into a clean conical flask and add dilute ammonium hydroxide drop by drop until bluish white precipitate is formed. Dissolve the ppt. by adding dilute acetic acid drop by drop and then add 1/4th t.t of acetic acid in excess. Add one t.t. of 10% potassium iodide solution and shake well. The liberated iodine titrated against standard sodium thiosulphate solution taken in a burette using starch as indicator towards the end point. (add starch when brown solution changes to light yellow). Disappearance of blue colour is

the end point. Note down the volume of hypo added and repeat the experiment for concordant values. Calculate the percentage of copper in the given sample of brass.

REACTIONS

$$Cu + 4HNO_3 \xrightarrow{heat} Cu(NO_3)_2 + 2H_2O + 2NO_2\uparrow$$
(reddish brown)

$$2Cu(NO_3)_2 + 4KI \rightarrow Cu_2I_2\downarrow + 4KNO_3 + I_2\uparrow$$
(brown)

$$2Na_2S_2O_3 + I_2 \longrightarrow 2NaI + Na_2S_4O_6$$ (sodium tetra thionate)

$$I_2 + \text{Starch} \longrightarrow \text{Starch-iodide complex (deep blue)}$$

RESULT

The percentage of Cu present in the given brass sample is ~~116.488%~~ 59.032%.

17/9/19

30/30

OBSERVATION AND CALCULATIONS

1. Preparation of brass solution:
Weight of brass taken is = 0.3659

2. Estimation of coppers:

Burette : Standard hypo (sodium thiosulphate) solution.

conical flask : 25cm³ brass solution + NH_4OH drop wise till a bluish white ppt is formed. Dissolve the ppt. by adding dilute acetic acid + 1/4th t.t of acetic acid in excess + one t.t. of 10% KI + shake well + one t.t. of distilled water.

Indicator : 2cm³ of freshly prepared starch.

End point : Disappearance of blue color.

Burette readings	I.	II.	III.
Final readings	6.8	6.8	6.8
Initial readings	0.0	0.0	0.0
volume of hypo run down in cm^3	6.8	6.8	6.8

$\therefore$ volume of hypo required $= V = 6.8\ cm^3$

NOTE

1. Normality of hypo = 0.05N
2. One gram equivalent weight of copper = 63.54

$1000 cm^3$ of 1N of hypo = one g equivalent weight of copper (63.54)

$\therefore V cm^3$ of 0.05N hypo $- X = \frac{V \times 0.05 \times 63.54}{1000}$

$= \frac{6.8 \times 0.05 \times 63.54}{1000}$

$X = 0.02160$ g of copper

$\therefore$ Weight of copper present in $25 cm^3$ of brass solution $= X = 0.02160$ g

$\therefore$ Weight of copper present in $250 cm^3$ of brass solution $= Y = \frac{X \times 250}{25}$

$$Y = \frac{0.0216 \times 250}{25}$$

$$Y = 0.2160g$$

∴ Percentage of copper in the given brass sample $= \frac{Y \times 100}{\text{weight of brass taken}(W)}$

$$= \frac{0.2160 \times 100}{0.3659}$$

$$= 59.032$$

∴ Percentage of copper in the given brass sample $= 59.032\%$.

DETERMINATION OF IRON IN A GIVEN SAMPLE OF HAEMATITE ORE SOLUTION

THEORY

Haematite is an important ore of iron containing mainly ferric oxide (Fe_2O_3) and small amount of silica (SiO_2). A known weight of the ore is digested with hydrochloric acid and the insoluble residue mainly containing silica is removed by filtration. The filtrate is diluted to 250 cm^3 and is supplied for the estimation. Iron is present in the solution as Fe^{3+} (ferric) ions and it is reduced to Fe^{2+} (ferrous) ions using stannous chloride ($SnCl_2$) in hot condition in presence of concentrated HCl. The excess of stannous chloride is oxidition by adding mercuric chloride solution. The ferrous ions in the resulting solution is titrated against standard potassium dichromate using potassium ferricyanide as external indicator. Potassium dichromate using ferricyanide produces an intense deep blue color with ferrous ions due to the formation of ferriferro-yanide complex.

Name of Experiment

Experiment No. 04

Date 24 09 19

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PROCEDURE:

a) Preparation of standard solution of potassium dichromate

Weigh given potassium dichromate crystals accurately using an electronic balance. Transfer the crystals of potassium dichromate into a clean 250cm³ standard flask using a funnel. Dissolve the crystals by adding distilled water and make up the solution to the mark by adding distilled water. Mix well for uniform concentration and calculate the normality of potassium dichromate solution.

b) Estimation of iron:

Pipette out 25cm³ of the haematite ore solution into a clean conical flask. Add ¼ t.t of concentrated hydrochloric acid and heat the solution nearly to boiling. Add stannous chloride drop by drop taken in a separate burette till yellow color solution becomes colorless and then add 6-8 drops of stannous chloride in excess. Cool the solution to lab temperature and add 10cm³ of saturated mercuric chloride at once, shake well and a silky white precipitate is formed. (if no ppt or black ppt is formed reject and repeat the experiment). add one t.t of distilled water and titrate this against standard potassium dichromate solution.

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Name of Experiment

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Experiment No 04

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taken in a burette, using potassium ferricyanide as external indicator. the end point is failure to observe blue colour when one drop of the titrated mixture is brought in contact with a drop of the indicator placed on paraffin paper. note down the volume of potassium dichromate added and repeat the experiment for concordant values. calculate the amount of iron in the given sample of hematite ore solution.

REACTIONS:

$$Fe_2O_3 + 6HCl \xrightarrow{Heat} 2FeCl_3 + 3H_2O$$

(haematite ore) (ferric chloride)

$$K_2Cr_2O_7 + 14HCl + 6FeCl_2 \longrightarrow 6FeCl_3 + 2KCl + 2CrCl_3 + 7H_2O$$

(ferrous chloride)

$$3FeCl_2 + 2K_3[Fe(CN)_6] \longrightarrow 6KCl + Fe_3[Fe(CN)_6]_2$$

(potassium ferricyanide) (ferriferro cyanide)

RESULT:

The amount of iron in the given Haematite ore solution is 1.165 g

24/9/19

29/30

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OBSERVATIONS AND CALCULATIONS

1. Preparation of standard potassium dichromate solution

Weight of weighing bottle + $K_2Cr_2O_7$ crystals = 5.0911 g
Weight of empty weighing bottle = 4.0074 g
∴ Weight of $K_2Cr_2O_7$ crystals taken = 1.0837 g

$$\text{Normality of } K_2Cr_2O_7 = \frac{\text{Weight of } K_2Cr_2O_7 \times 4}{\text{Equivalent weight of } K_2Cr_2O_7}$$

$$N_{K_2Cr_2O_7} = \frac{1.0837 \times 4}{49} = 0.0884\,N(a)$$

2. Estimation of iron:

Burette: Standard $K_2Cr_2O_7$ solution.

Conical flask: 25 cm^3 of haematite ore solution + 1/4 t.t concentrated HCl + heat + $SnCl_2$ drop wise till yellow color changes to colorless + 5-6 drops of $SnCl_2$ in excess + cool the solution to room temperature + 10 cm^3 of saturated $HgCl_2$ + shake well + one t.t of distilled H_2O

Indicator: Potassium ferri cyanide as external indicator.

End point: Failure to observe blue color; when a drop of reacting mixture is brought in contact

with the indicator placed on paraffine paper.

Burette readings	Pilot	1	2	3
Final readings		5.9	5.9	5.9
Initial readings		0.0	0.0	0.0
Volume of $K_2Cr_2O_7$ run down in cm^3s		5.9	5.9	5.9

volume of $K_2Cr_2O_7$ required $= V = \underline{5.9}\ cm^3$

NOTE: Equivalent weight of iron = 55.85g

1000 cm^3 of 1N of $K_2Cr_2O_7$ = one gm equivalent weight of iron (55.85g)

$\therefore V cm^3$ of (a) $N_{K_2Cr_2O_7} = X = \frac{(a) \times V \times 55.85}{1000}$

$$X = \frac{(0.0884)(5.9)(55.85)}{1000}$$

$X = \underline{0.0291}$ gm of iron

$\therefore$ Weight of iron present in 25 cm^3 of haematite solution $= X = \underline{0.0291}$ g

$\therefore$ weight of iron present in 1000 cm^3 of Haematite ore solution $= Y = \frac{X \times 1000}{25}$

Name of Experiment

Experiment No.

$y = \underline{1.165}\ g$

Name of Experiment

Date 01 10 19

Experiment No. 05

Page No. 1 2

DETERMINATION OF CHEMICAL OXYGEN DEMAND (COD) OF THE GIVEN INDUSTRIAL WASTE WATER SAMPLE

THEORY:

The chemical oxygen demand (COD) test is extensively employed for the measurement of the pollution strength of industrial wastes. Chemical oxygen demand is a measure of the total quantity of oxygen required for oxidation of organic compounds of wastes to CO_2 and water by a strong oxidizing agent. This parameter is particularly valuable in surveys designed to determine the control losses to sewer systems. Results may be obtained within a relatively short time and measures taken to correct error on the day they occur.

Waste water contains organic impurities which include straight chain aliphatic and aromatic compounds, such as alcohols, acids, amines, pyridine and other oxidizable materials. They oxidize more effectively when silver sulphate is added as a catalyst. But silver sulphate reacts with chlorides in the waste water to form precipitates which are oxidized partially by this procedure. This difficulty is overcome by

adding mercuric sulphate to the sample.

PROCEDURE:

1. Preparation of standard mohr's salt or ferrous ammonium sulphate (FAS) solution:

Weigh given Mohr's salt crystals accurately using an electronic balance and transfer the crystals into a clean 250cm³ standard flask. Dissolve the crystals by adding two t.t. of dilute sulphuric acid and little distilled water. and make it up to the mark by adding distilled water. Mix well for uniform concentration and calculate the normality of Mohr's salt solution.

2. Determination of COD of waste water:

Pipette out 25cm³ of the industrial waste water and exactly 25cm³ of standard potassium dichromate solution (0.1N) into a clean conical flask add one tt of 1:1 sulphuric acid and 2 to 4 drop of ferroin indicator and titrate against standard Mohr's salt solution taken in a burette untill blue-green color changes to reddish-brown. Note down the volume of Mohr's salt solution added for unreacted potassium dichromate solution (V₁) and repeat the experiment to get concordant values. using

blank titre value determines the volume of potassium dichromate required to oxidize organic compounds and calculate the COD of the given industrial waste water.

3. Blank titration

Add exactly 25cm³ of standard potassium dichromate solution into a clean conical flask and one ml of 1:1 sulphuric acid. Add 2-4 drops of ferrion indicator and titrate this against Mohr's salt solution taken in a burette until blue-green colour changes to reddish brown. Note down the volume of Mohr's salt solution required for blank titration(V_2).

Reactions:

Industrial wastewater (organic compounds) + oxidising agent ($K_2Cr_2O_7$) $\xrightarrow[H_2SO_4]{heat}$ $H_2O + CO_2\uparrow$

$$K_2Cr_2O_7 + 7H_2SO_4 + 6FeSO_4 \longrightarrow 3Fe_2(SO_4)_3 + K_2SO_4 + Cr_2(SO_4)_3 + 7H_2O$$

ferric ion indicator complex (Fe^{3+}) (blue green) $\underset{K_2Cr_2O_7}{\overset{FAS}{\rightleftharpoons}}$ ferroin indicator (Fe^{2+}) (reddish brown)

Result

The COD of the give waste water sample contains 378.9mg of oxygen

01/10/19

30/30

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OBSERVATIONS AND CALCULATIONS

1. Preparation of Mohr's salt or ferrous ammonium sulphate (FAS) solution:

Weight of weighing bottle + FAS crystals = 13.0082 g
Weight of empty weighing bottle = 3.8695 g
∴ Weight of FAS crystals taken = 9.1387 g

$$\text{Normality of FAS} = \frac{\text{Weight of FAS} \times 4}{\text{Equivalent weight of FAS}}$$

$$N_{FAS} = \frac{9.1387 \times 4}{392}$$

$$N_{FAS} = 0.09325 \text{ (a) N}$$

2. Estimation of chemical oxygen Demand (back titration)

Burette : standard FAS solution.

Conical flask : 25 cm^3 of waste water + 25 cm^3 of standard $K_2Cr_2O_7$ solution + one tt of 1:1 H_2SO_4.

Indicator : 4-6 drops of ferroin indicator.

End point : Blue green to reddish brown color

Burette readings	I	II	III
Final readings	7.8	7.8	7.8
Initial readings	0.0	0.0	0.0
Volume of FAS run down in cm^3	7.8	7.8	7.8

∴ volume of FAS solution required $= V_1 = 7.8 cm^3$

Blank titration or (FAS >< $K_2Cr_2O_7$)

Burette : Standard FAS solution.

Conical flask : $25 cm^3$ of $K_2Cr_2O_7$ solution + 1 tt of 1:1 H_2SO_4

Indicator : 4-6 drops of Ferrion indicator

End point : Blue green to reddish brown color.

∴ volume of FAS solution required for blank titration

$= V_2 =$ Final B.R − Initial B.R $= \underline{20.5}$

$V_2 = \underline{20.5 cm^3}$

∴ volume of $K_2Cr_2O_7$ required for the oxidation of organic compounds of waste water $= V_2 - V_1 = \underline{20.5 - 7.8}$

$= \underline{12.7 cm^3}$

$1000 cm^3$ of 1N of FAS $= 1000 cm^3$ of 1N $K_2Cr_2O_7$ solution

$= 8g$ of oxygen

$V_2 - V_1$ cm³ of (a)N FAS = $X = \frac{8 \times (a) \times V_2 - V_1}{1000}$

$$x = \frac{8(0.09325)(12.7)}{1000}$$

$$x = 9.474 \times 10^{-3} g \text{ of oxygen}$$

∴ Amount of O_2 required to oxidise organic compounds present in 25cm³ of waste water $= x = 9.47 \times 10^{-3} g$

∴ Amount of O_2 required to oxidise organic compounds present in 1000cm³ of waste water $= Y = \frac{X \times 1000}{25}$

$$= 378.96 \times 10^{-3} g$$

$$Y = 0.378 g$$

∴ COD of waste water $= Y \times 1000$

$$= 0.3789 \times 1000 mg$$

$$= 378.9 mg$$

∴ COD of waste water = 378.9 mg of oxygen

DETERMINATION OF Pka VALUE OF A WEAK ACID USINGS pH METER.

Theory:

The strength of an acid is experimentally measured by determining its equilibrium constant or dissociation constant (Ka). Strong acids are completely ionized in aqueous solution and hence it is impossible to determine the dissociation constant of strong electrolytes. But in case of weak electrolytes are partially ionized in aqueous solution and hence the equilibrium constant of weak electrolytes can be experimentally determined.

Let us consider weak acid like formic acid, which ionizes, freely in aqueous solution as,

$$HCOOH_{(aq)} + H_2O_{(l)} \xrightleftharpoons{Ka} H_3O^+_{(aq)} + HCOO^-_{(aq)}$$

$$\therefore Ka = \frac{[HCOO^-][H_3O^+]}{[HCOOH]}$$

Pka is a modern method of expressing the strength of weak acid, i.e., $Pka = -\log_{10} Ka$

Pka value is determined by measuring the changes in pH of acid solutions at different amount of the base added. In the titration of an acid with a

the PH of the solution rises gradually at first and then more rapidly. At the equivalence point there is a very sharp increase in PH for a very small quantity of base added. Beyond the equivalence point, the PH increases slightly on addition of base. The titration curve is obtained by plotting changes in PH against different volume of the base added and from the graph, we obtain the equivalence point of an acid.

The Pka value of an acid can be calculated using Henderson equation,

$$PH = Pka + \log_{10} \frac{[salt]}{[Acid]}$$

At half equivalence point [salt] = [acid] and hence PH at half equivalence point gives the Pka value of weak acid i.e PH = Pka

PROCEDURE:

Transfer given quantity of weak acid (25cm³) into a clean 100cm³ beaker followed by adding one t.t of distilled water.

Switch on the PH meter and adjust the temperature knob to the lab temperature. Connect the combined electrode (i.e glass and calomel electrode) to the PH meter. Wash the electrode with distilled water and immerse the electrode into the acid taken in

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a micro burette in to the acid, stir the solution using magnetic stirrer and measure the pH of the acid, stir the solution using magnetic stirrer and measure the pH of the acid and continue the addition of sodium hydroxide until the sudden raise in the pH. then take few more readings and plot the graph pH against the volume of sodium hydroxide added (graph NO.1). Take the difference in the pH of acid (ΔpH) and the volume of sodium hydroxide added (ΔV). Plot the graph, $\Delta pH/\Delta V$ against the volume of sodium hydroxide added (graph No.2) and from the graph 2, determine the equivalence point of the acid Determine the half equivalence point that gives the Pka value of weak acid by the graph 1.

Note: Switch off the instrument after experiment is Completed.

Results:

1) Equivalence point 4.0
2) Half E.P 2.0
3) PH of EP 8.7
4) PH at H.E.P 5.2
5) Pka of given acid 5.2

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OBSERVATIONS AND CALCULATIONS

Volume of NaOH (cm^3)	pH	ΔpH	ΔV	$\Delta pH/\Delta V$
0.0	4.31			
0.5	4.6	0.3		0.6
1.0	4.8	0.2		0.4
1.5	5.0	0.1		0.3
2.0	5.2	0.2		0.4
2.5	5.4	0.1		0.3
3.0	5.6	0.2	0.5	0.5
3.5	6.0	0.4		0.8
4.0	8.7	2.6		5.2
4.5	10.1	1.4		2.8
5.0	10.6	0.4		0.9
5.5				

Results:

1) Equivalence point = 4.0

2) Half E.P = 2.0

3) pH at E.P = 8.7

4) pH at H.E.P = 5.2

5) pKa of given acid = 5.2

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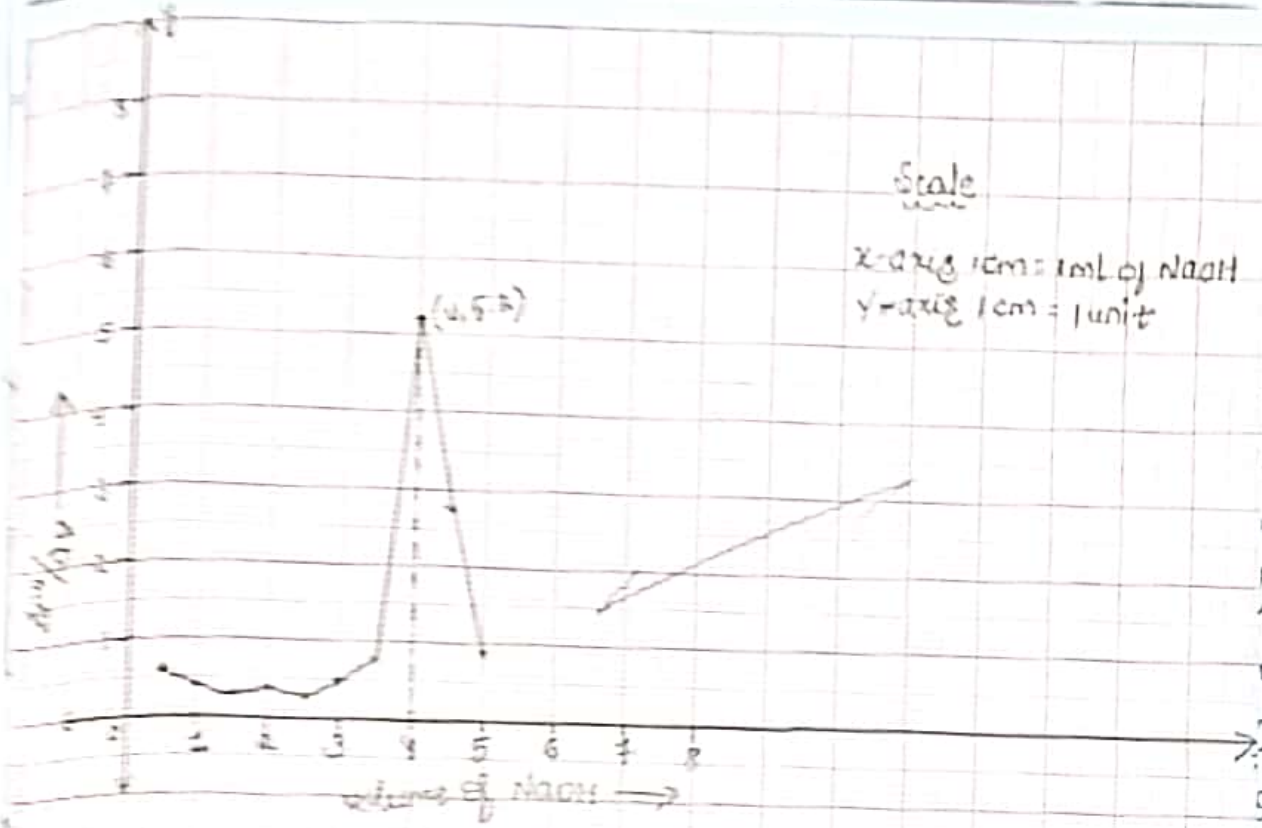

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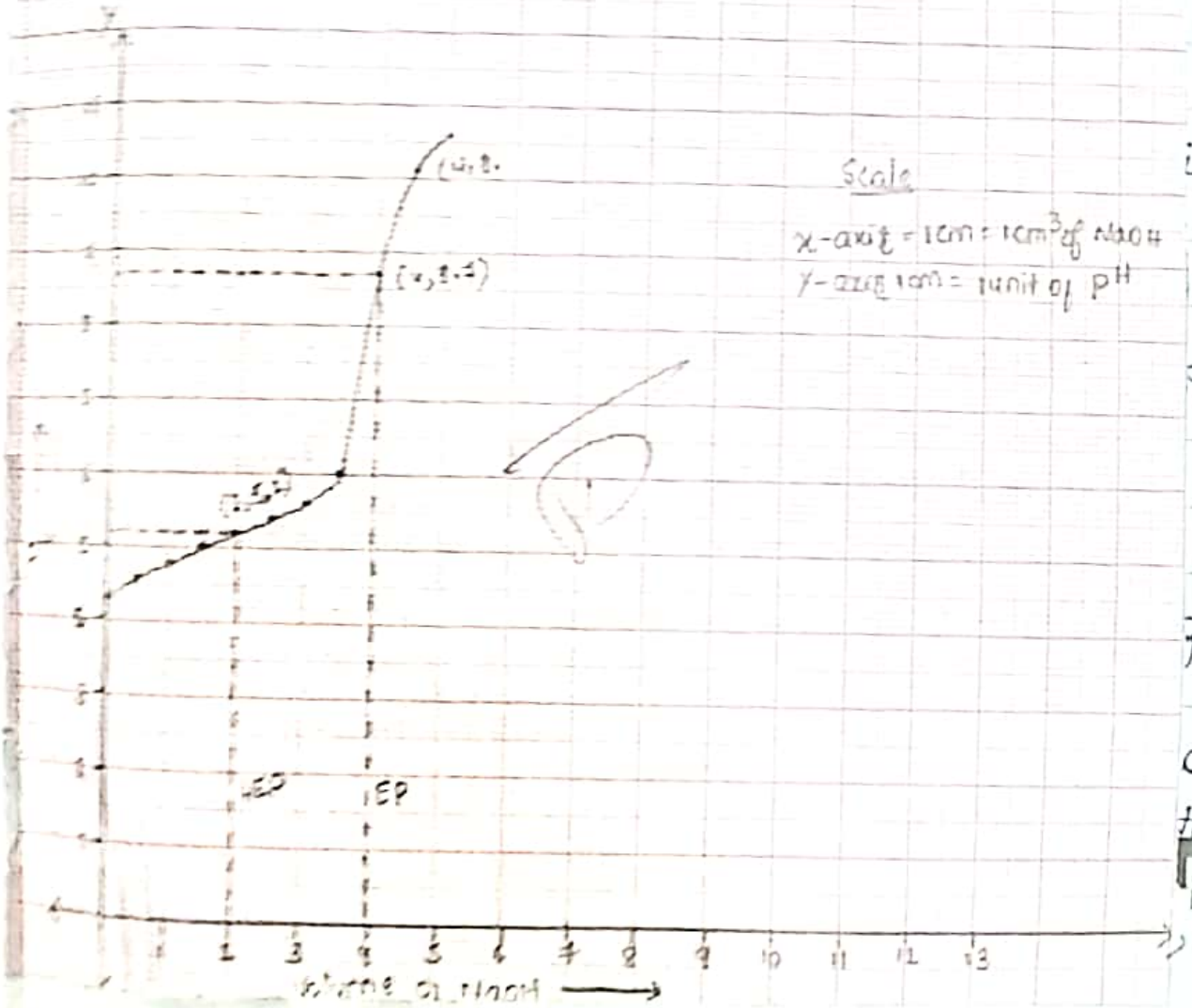

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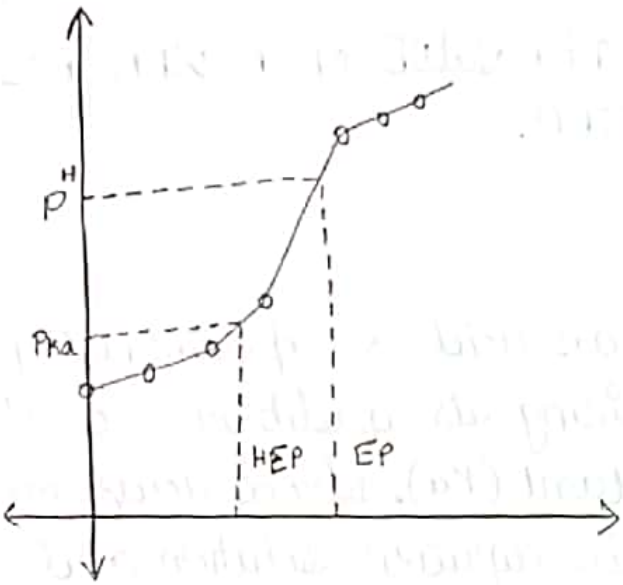


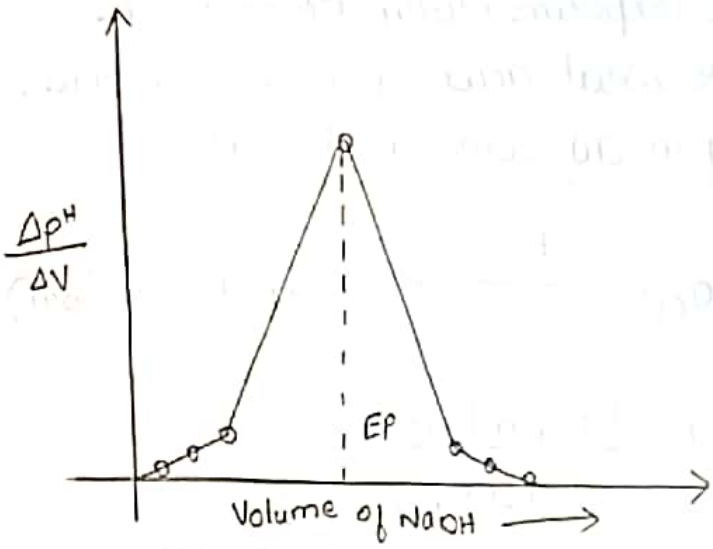
Scale:

X-axis, 1.0cm = 1 cm^3

Y-axis, 1.0cm = 1 pH

graph No 1

Volume of NaOH added in cm^3



Scale:

x-axis, 1.0cm = 1 cm^3

Y-axis 1.0cm = 1 units

graph - 2

Determination of viscosity coefficient of given liquid using Ostwald's viscometer.

Theory:

Viscosity arises due to internal friction between moving layers of molecules. A liquid flowing through a cylindrical tube of uniform diameter is expected to move in the form of molecular layers. A layer close to the surface is almost stationary while that at the axis of tube moves faster than any other intermediate layer. A slow moving layer exerts a drag or friction on its nearest moving layer backwards. This property of a liquid by which it retards or opposes motion of a liquid between the layers is called viscosity. The co-efficient of viscosity is defined as the tangential force per unit area required to maintain a unit velocity gradient between any two successive layers of a liquid situated unit distance apart. The coefficient of viscosity of a liquid is given by poiseuille's formula,

$$\eta = \frac{\pi P r^4 t}{8 V l}, \text{ where } P = h d g$$

$$\eta = \frac{\pi h d g r^4 t}{8 V l}$$

where η is the coefficient of viscosity of the liquid
V = Volume of the liquid, r = radius of the tube

l = length of the tube, P is the pressure difference between the two ends of the tube, h = height of the capillary tube, d = density of the liquid, and g = gravit-ational force. If equal volumes of two different liquids are allowed to flow through the same tube under identical conditions, then,

$$\frac{\eta_w}{\eta_L} = \frac{t_w d_w}{t_L d_L}$$

where, η_w = co-efficient of viscosity of the standard liquid (water), and η_L = co-efficient of viscosity of the give liquid which can be determined. t_w = time taken by the standard liquid (water), t_L = time taken by the given liquid. viscosity is a temperature dependent property and is inversely proportional to temperature (T), i.e. $\eta \propto 1/T$. Hence the measurments are carried out at constant temperature.

Procedure:

1) clean the given viscometer with distilled water and rinse with acetone Dry it in an electric oven and then fix the viscometer vertically to a stand.

2) Transfer a known volume (say 15 cm^3) of distilled water taken in a burette into the viscometer. Suck the distilled water above the upper mark 'A' in the

and allow it to flow freely through a capillary. Start the stop clock when the water level just crosses the upper mark 'A' and stop it when the water level just crosses the lower mark 'B' in the viscometer. Note down the time of flow of water in seconds (t_w) and repeat the experiment to get agreeing values.

3) Pour out the distilled water and rinse the viscometer with acetone again. Dry it and fix the viscometer vertically to a stand.

4) Transfer the known volume (say 15cm^3) of the given organic liquid into a viscometer. Note down the time of flow of organic liquid (t_L) in seconds as similar to the water and repeat the experiment to get agreeing values.

5) Determine the viscosity co-efficient of the given organic liquid using the relation,

$$\eta_L = \frac{\eta_w t_L d_L}{t_w d_w}$$

and the given values of η_w d_w and d_L

Result

$\therefore \eta_L$ = co-efficient of viscosity of liquid = 30.444 milli Poise

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Observations And calculations

Trial No	I	II	III	Average time in sec
Time taken by water in sec (t_w)	39	39	39	39
Time taken by liquid in sec (t_L)	141	143	144	142.6

$$\eta_L = \frac{\eta_w t_L d_L}{t_w d_w}$$

Note: Lab temperature = _____ °C

where t_w = time taken by water = 39 sec

t_L = time taken by liquid = 142.6 sec

d_w = density of water = 0.9959 g/cc

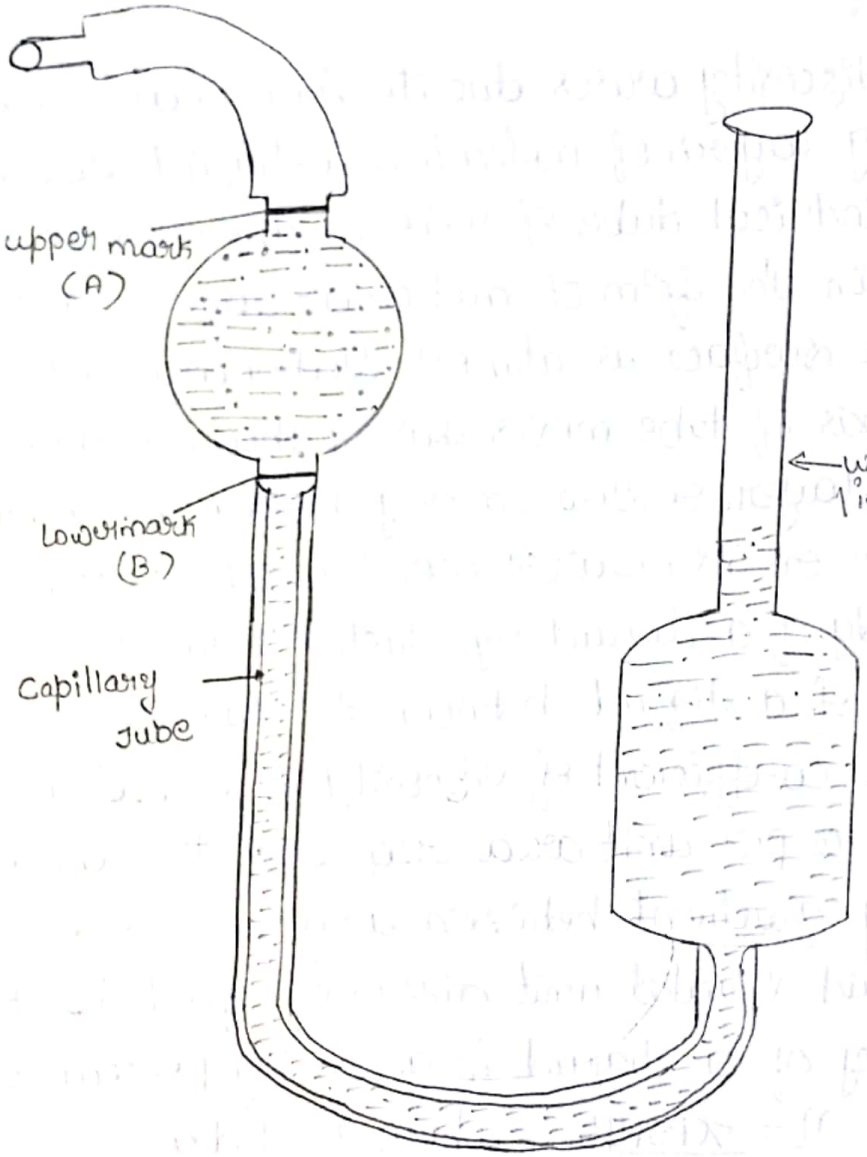
d_L = density of liquid = 1.0137 g/cc

η_w = co-efficient of viscosity of water = 8.18 millipoise

η_L = co-efficient of viscosity of liquid = 30.444 millipoise

$$\eta_L = \frac{\eta_w t_L d_L}{t_w d_w} = \frac{8.18 \times 142.6 \times 1.0137}{39 \times 0.9959}$$

$\therefore \eta_L$ = co-efficient of viscosity of liquid = 30.444 milli poise



POTENTIOMETRIC ESTIMATION OF FAS USING STANDARD $K_2Cr_2O_7$ SOLUTION

Theory:

When the electrode is dipped in the solution, the potential of an electrode depends on the concentration of the ions in the electrolyte. Therefore determination of maximum potential can serve as an indicator in potentiometric analysis. Determination of change of potential with concentration gives more accurate results than volumetric analysis.

The given electrode is combined with saturated calomel electrode and dipped in electrolyte, an electro chemical cell is formed. The emf of this cell is measured using potentiometer. During the titration the change in emf of the cell or concentration of ions in the electrolyte can be mesured. Then $\Delta E/\Delta V$ is plotted against the volume of the titrant there will be a peak corresponding to the end point of the reaction.

∴ Emf of the cell is calculated using Nernst equation,

$$E_{cell} = E^0_{cell} + \frac{0.0591}{n} \log \frac{[Products]}{[Reactants]}$$

where E_{cell} = emf of the cell, E^0_{cell} standard emf of the cell.

n = number of electrons involved in the reaction

[products] = concentration of the products and

[Reactants] = concentration of the reactants.

Transfer the given amount of Mohr's salt solution (25cm3) in to a clean 100cm3 beaker. to that add one test tube of dilute sulphuric acid.

Switch on the potentiometer and adjust the temperature knob to the lab temperature. Wash the electrodes with distilled water and immerse the electrodes in to the Mohr's salt solution taken in a beaker. Measure the emf of the solution in terms of mV. Add 0.5cm3 of potassium dichromate solution at a time taken in a micro burette in to the Mohr's salt solution. Stir the solution and measure the emf of the solution and continue the addition potassium dichromate added until the sudden raise in emf. Then take few more readings and take the difference in the emf of solution (ΔE) and the volume of potassium dichromate added (ΔV). Plot the graph, $\Delta E/\Delta V$ against the volume of potassium dichromate added. From the graph, $\Delta E/\Delta V$

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determine the volume of potassium dichromate required to rapid increase in the emf of the solution. calculate the amount of Mohr's salt given in the solution.

$$K_2Cr_2O_7 + 7H_2SO_4 + 6FeSO_4 \longrightarrow 3Fe_2(SO_4)_3 + K_2SO_4 + Cr_2(SO_4)_3 + 7H_2O$$

Result

Amount of Mohr's salt present in the given solution is 7.050 g

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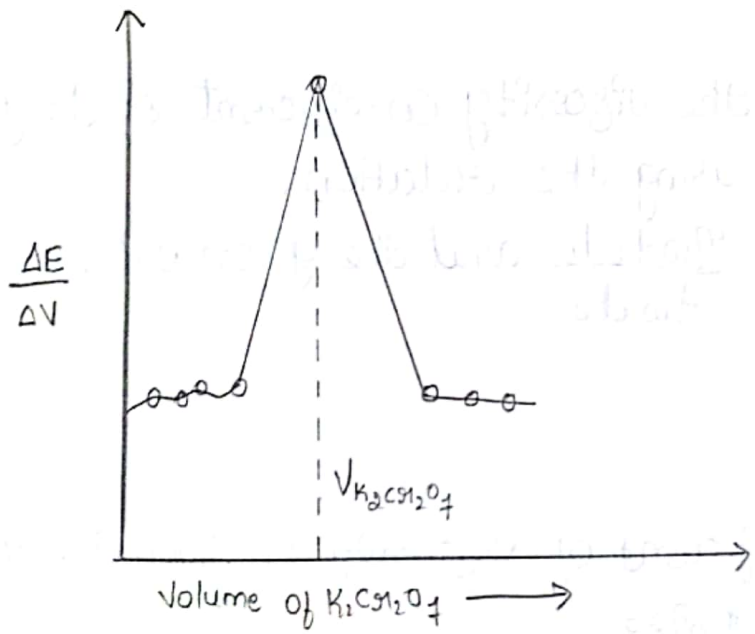
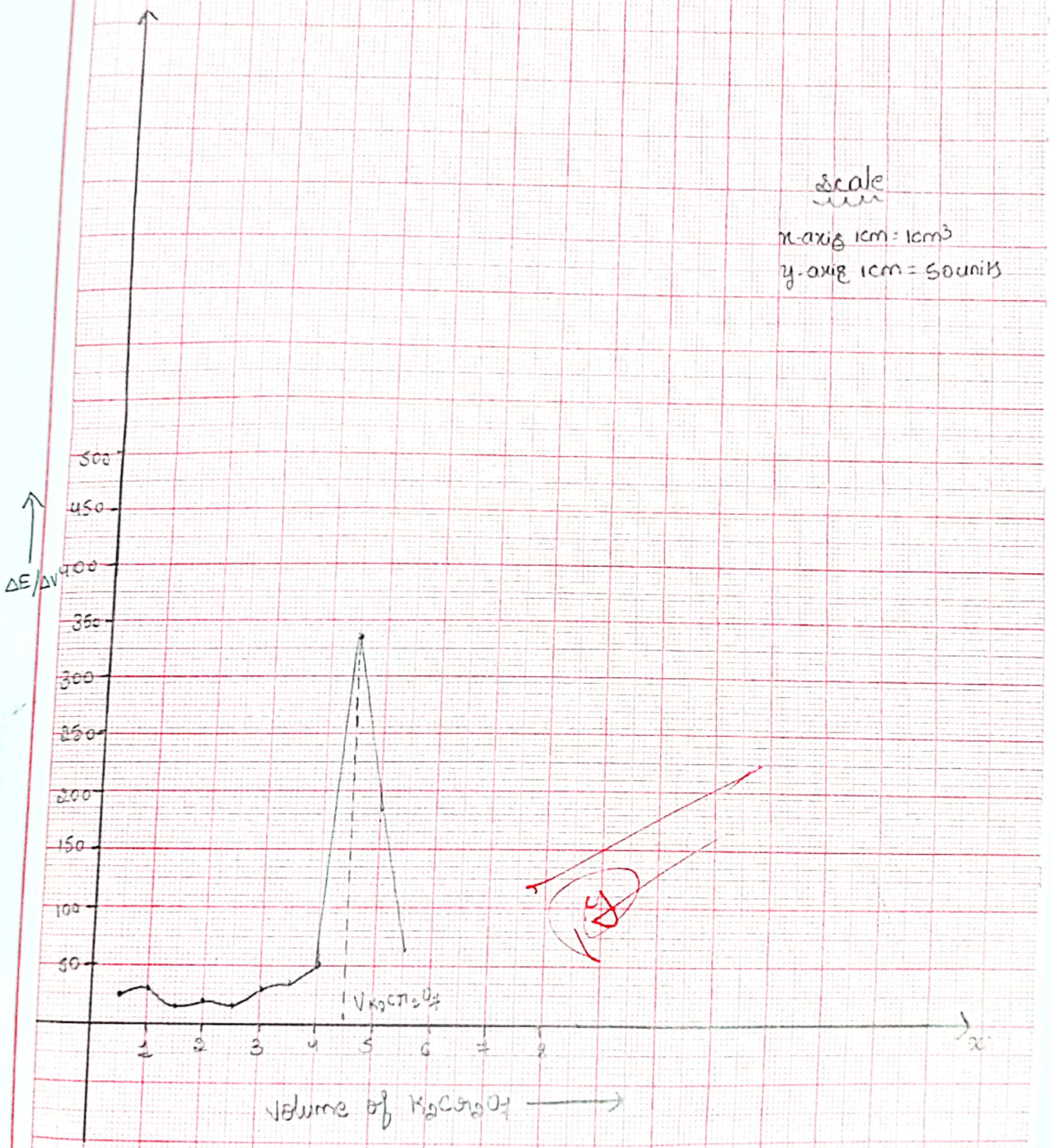
Volume of $K_2Cr_2O_7$	EMF(E) in mV	ΔE	ΔV	$\Delta E/\Delta V$
0.0	421			
0.5	434	13		26
1.0	448	14		28
1.5	458	10		20
2.0	470	12		24
2.5	477	7		14
3.0	491	14	0.5	28
3.5	509	18		36
4.0	537	28		56
4.5	720	183		366
5.0	813	93		186
5.5	847	34		68



Scale
x-axis 1.0cm = 1cm^3
y-axis 1cm = 50units


Scale
x-axis 1cm = 1cm3
y-axis 1cm = 50units
ΔE/ΔV
500
450
400
350
300
250
200
150
100
50
1
2
3
4
5
6
7
8
x
V K2Cr2O7 = 0.4
Volume of K2Cr2O7

Note:

1. Normality of $K_2Cr_2O_7$ = $N_{K_2Cr_2O_7} = 0.1N$
2. Equivalent weight of Mohr's salt = 392

∴ Normality of Mohr's salt solution = $\frac{N_{K_2Cr_2O_7} \times V_{K_2Cr_2O_7}}{V_{Mohr's\ salt}}$

$$N_{Mohr's\ salt} = \frac{4.5 \times 0.1}{25} = 0.018N$$

Weight per liter of Mohr's salt crystals = X =

$N_{Mohr's\ salt}$ X Equivalent weight of salt

$X = 0.018 \times 392$

$X = 7.056g$

∴ Amount of Mohr's salt present in the given solution is = 7.050g.

Result: Amount of Mohr's salt present in the given solution 7.050g

CONDUCTOMETRIC ESTIMATION OF ACID MIXTURE USING STANDARD SODIUM HYDROXIDE

Theory:

Measurement of conductance can be employed to determine the end point in acid-base titrations. In conductmetric titrations, there is a sudden change in conductance of the solution near the end point. The end point is determined graphical-y by plotting conductance (ordinate) against sodium hydroxide (abscissa)

Conductometeric titration may be applied for the determination, of acid and acids pressent in a mixture. In the titration of a mixture of acid with a strong base (sodium hydroxide), the conductance falls until the strong acid is neutralized completely, and the weak acid undergo dissociate and gets neutralized. This results in the increases in conductance of the solution as the weak acid is consumed and converted into salt. when the neutralization of second acid is complete, there is a steep increase in conductance due to ions provided by the strong base.

Procedure:

Transfer the given acid mixture ($25cm^3$) into a clean $100cm^3$ beaker. Fill standard NaOH (0.2N) solution into $10cm^3$ burette. Place the conductivity cell inside the beaker, so that the electrodes are immersed completely in the acid solution. Connect the cell to the conductometer and measure the conductance. Add $0.5cm^3$ of NaOH each time, till there is a rise in conductance. After each addition stir the solution and note down the corresponding decrease in the conductivity of the solution. As the titration proceeds the conductance is gradually a decreases and then increases slowly and finally increases sharply. Take 5-6 readings when conductance begins to increase sharply. Plot a graph of conductance against volume of NaOH added.

From the graph determine the volume of NaOH required be $V_1 cm^3$ for strong acid as the titration proceeds, the second neutralization point V_2 will be obtained. The difference $V_2 - V_1$ will give the amount of base neutralization for weak acid. Calculate the weight per litter of HCl and CH_3COOH

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

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

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Result:

a) The weight of HCl/dm³ = 0.5548 g

b) The weight of CH_3COOH/dm³ = 1.9696 g

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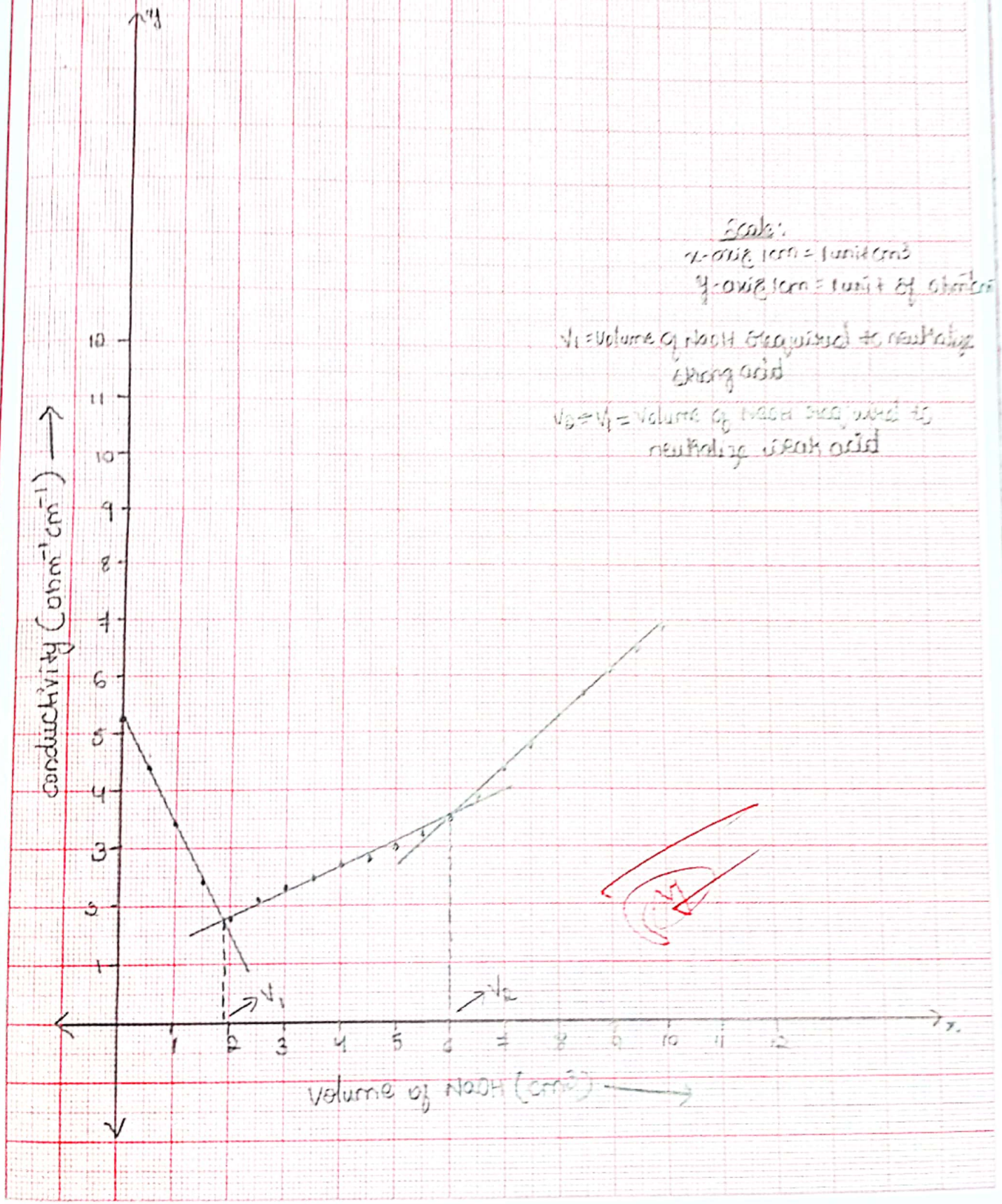
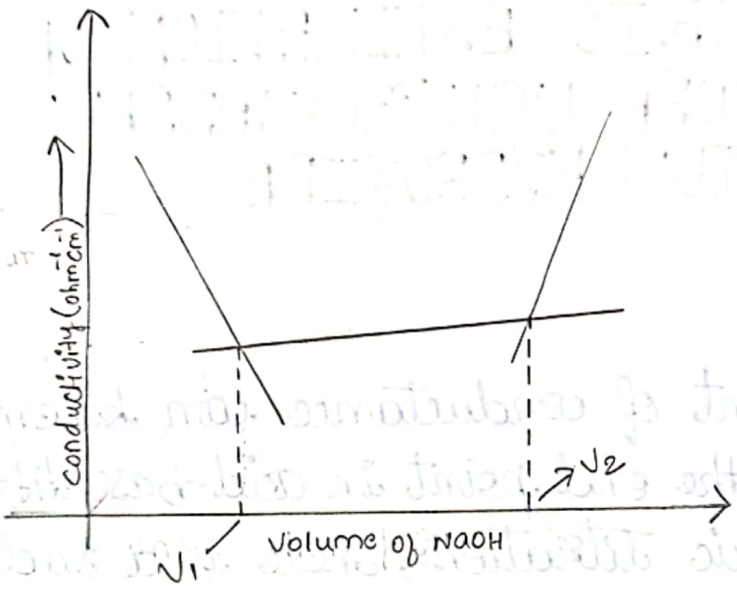
Observations AND calculations

Estimation of acid mixture

vol of NaOH in cm^3	conductance $ohm^{-1}cm^{-1}$	vol. of NaOH in cm^3	conductance $ohm^{-1}cm^{-1}$
0.0	5.52	5.5	3.25
0.5	4.40	6.0	3.50
1.0	3.41	6.5	3.95
1.5	2.43	7.0	4.44
2.0	1.89	7.5	4.88
2.5	2.1	8.0	5.33
3.0	2.31	8.5	5.76
3.5	2.51	9.0	6.16
4.0	2.70	9.5	6.53
4.5	2.88	10.0	6.92
5.0	3.06	10.5	





Scale :
x-axis 1cm = 5mg/cm³
Y-axis 1cm = 0.05 OD

Estimation of HCl

Volume of NaOH required to neutralize HCl = V_1 = 1.9 cm³

Normality of HCl = $\frac{N_{NaOH} \times V_1}{25}$

$= \frac{0.2 \times 1.9}{25} = 0.0152 N$

weight per liter of HCl = X = N_{HCl} × Equivalent weight of HCl

$= 0.015 \times 36.5$

$X = 0.5548 g$

Estimation of CH_3COOH

Volume of NaOH required to neutralize CH_3COOH = $(V_2 - V_1)$
= 4.1 cm³

Normality of CH_3COOH = $\frac{N_{NaOH} \times V_{(V_2-V_1)}}{25}$

$= \frac{0.2 \times 4.1}{25} = 0.0328 N$

Weight per liter of $CH_3COOH = X = N_{CH_3COOH} \times$ Equivalent weight of CH_3COOH

$= 0.0328 \times 60.05$

$X = 1.969649$

DETERMINATION OF COPPER BY COLORIMETRIC METHOD

Theory:

When a monochromatic light of intensity I_0 is incident on a transparent medium, a part of light is absorbed by the media (I_a), a part of light is reflected (I_r) and the remaining part of light is transmitted (I_t).

$\therefore I_0 = I_a + I_t + I_r$ (for a glass-air interface, I_r is negligible)

$\therefore I_0 = I_a + I_t$

The ratio of $I_t/I_0 = T$ called transmittance and $\log I_0/I_t = A$ which is called absorbance or optical density. The relationship between absorbance (A), concentration (c) (expressed in mol/dm^3) and path length (t) (expressed in cm) is given by Beer-Lambert's law.

$$A = \log I_0/I_t = \varepsilon c t.$$

where ε is the molar extension coefficient which is a constant for a given substance at a given wave length and t is the path length of the cell tube which is also constant for given cell tube. Therefore, $A \propto c$. Hence plot the graph, absorbance against concentration of the solution gives a straight line.

A series of standard solution of copper salt and test solution is treated with ammonia to get blue cuprammonium complex and is diluted to a definite volume. The absorbance of each of these solution and test solution is measured at 620nm since the complex shows maximum absorbance at this wavelength. The absorbance values are plotted against concentration of copper get a calibration curve and concentration of copper in test solution is determined from the graph.

Procedure :

Transfer the given copper sulphate solution (stock solution) to a burette and draw out 5, 10, 15, 20 and 25cm³ of the solution into 50cm³ standard flasks. Add 5cm³ of 1:1 ammonia solution into a 50 cm³ standard flask make up the solution to the mark with distilled water & mix well for uniform concentration

Switch on the colorimeter & warm it for 5minutes. Using the filter knob(B), select the wave length of $\lambda max = 620$ nm for copper sulphate solution. Take the blank solution in the cell tube & keep it in the colorimeter and adjust zero absorbance for 100% transmittance using the knob (A). The measure the optical density or absorbance for each standard solution & test solution

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using a plotting optical density or absorbance against the copper in test solution & calculate the volume of copper sulphate in the given test solution.

$CuSO_4 + 4NH_3 \longrightarrow [Cu(NH_3)_4SO_4]$ (blue color)

[cuprammonium sulphate]

Preparation of blank solution :-

Blank solution can be prepared by adding $5.0 cm^3$ of 1:1 ammonia & distilled water in $50 cm^3$ standard flask. Mix well for uniform concentration.

Result

1. The volume of test solution given — $7.5 cm^3$
2. Absorbance of test solution — 0.10
3. concentration of copper in test solution — $7.5 mg/cm^3$
4. concentration of copper sulphate solution — $29.47 g/cm^3$

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Observations and Calculations

Volume of copper sulphate in cm^3	Concentration of copper sulphate in mg/cm^3	Concentration of copper in mg/cm^3	Absorbance (A) or OD at 620nm
5	$3.93 \times 5 = 19.65$	$5 \times 1 = 5$	0.06
10	$3.93 \times 10 = 39.30$	$10 \times 1 = 10$	0.15
15	$3.93 \times 15 = 58.95$	$15 \times 1 = 15$	0.21
20	$3.93 \times 20 = 78.60$	$20 \times 1 = 20$	0.28
25	$3.93 \times 25 = 98.25$	$25 \times 1 = 25$	0.35
Test solution	$3.93 \times 0.10 = 29.475$	$7.5 \times 1 = 7.5$	0.10

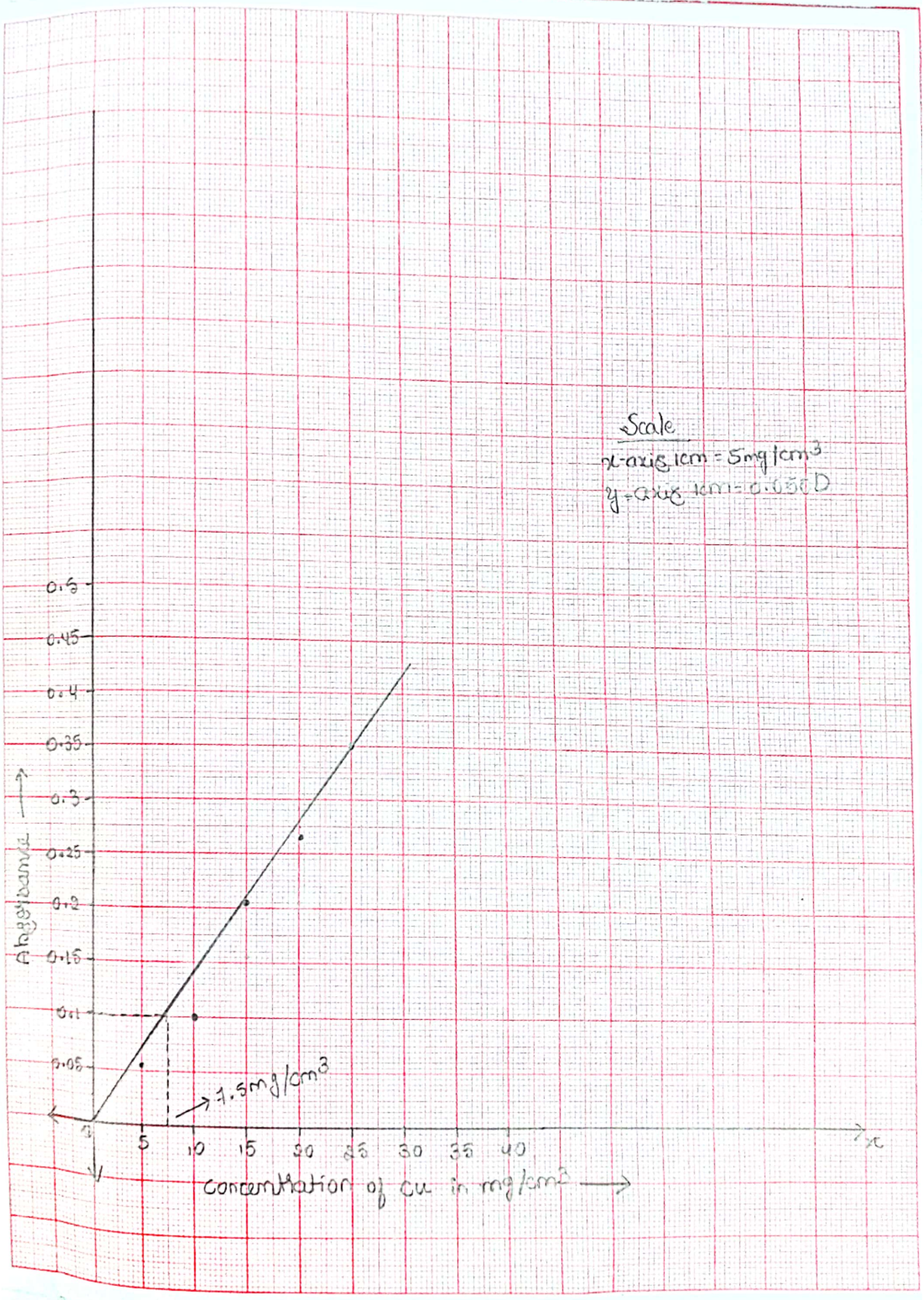
Note: Molecular weight of copper sulphate pentahydrate $= 249.54$

1. Atomic weight of copper $= 63.54$.

$1.0 cm^3$ of stock solution $=$ 3.93 mg of copper sulphate

249.54g of $CuSO_4 \cdot 5H_2O$ = 63.54g of copper

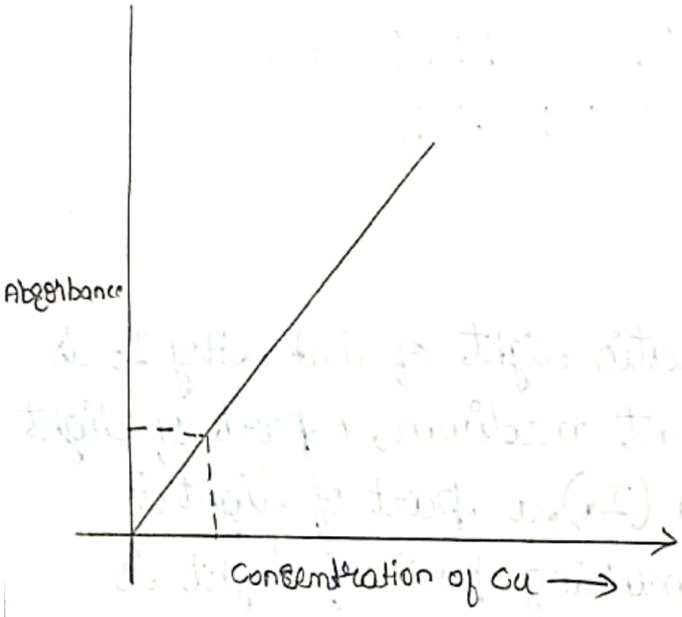
$\therefore$ 3.93mg of $CuSO_4 = \frac{63.54 \times 3.93}{249.54} = 1.0$mg of copper.



Scale:

x-axis, 1.0cm = 5mg/cm^3

y-axis, 1.0cm = 0.000D



∴ 1cm^3 of $CuSO_4$ = 3.93mg of $CuSO_4$ = 1.0mg of copper.

C = concentration of copper in test solution = 7.5 mg/cm^3

V cm^3 of copper sulphate = C mg of copper.

∴ Vcm^3 of copper sulphate = $V = \frac{C}{1.0} = \frac{7.5}{1.0} = 7.5\,cm^3$

Hence the volume of test solution given = V = 7.5 cm^3