|| 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.....KEERTHANA...V..... USN.....19CSE036..... has satisfactorily completed the course of experiments in ..Engineering....chemistry..... Laboratory (Course Code.....18CHEL16.....) prescribed by the Adichunchanagiri University, for .....I..... Semester, BE...Computer..Science...and. Engineering, of this College in the year 2019 - 2020

Record Marks : 30 Test Marks : 10

IA Marks : 30+10=40

Date : 10/12/19

Staff Incharge

Head of the Department

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Name of the Student : Keerthana.V Class : 1st year Sem. I Sem

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02	03/09 2019	Determination of calcium oxide in the given sample of cement solution by rapid EDTA method	5-7	5	10	5	10	30	[illegible]
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$\frac{296}{300}$ $\frac{30}{30}$

Name of Experiment

Experiment No.

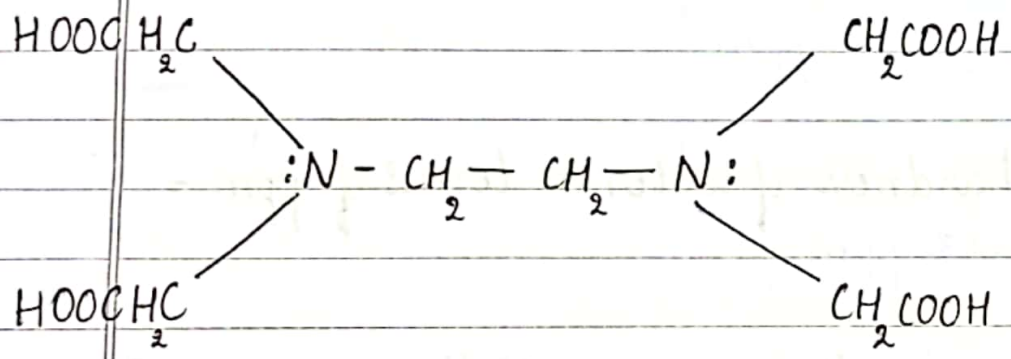
Experiment no. :- 1

Determination of Total Hardness of Water.

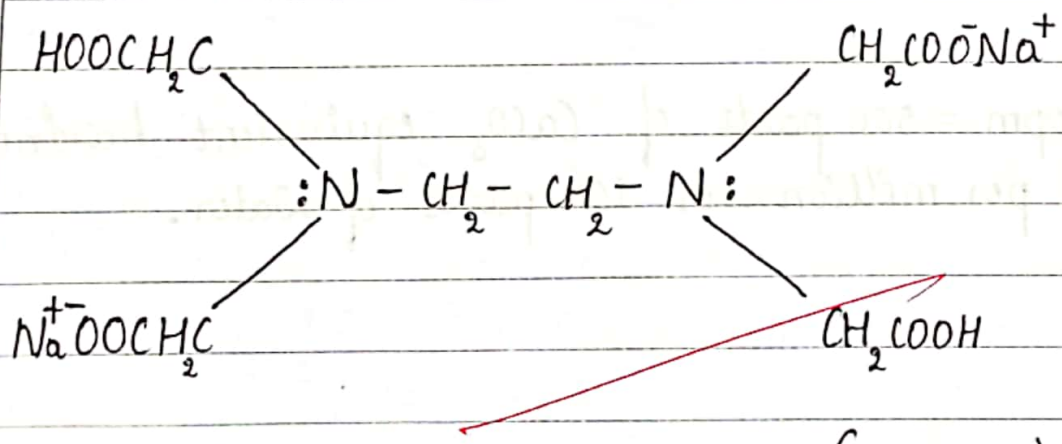
Theory :

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 (ie, parts per million) of $CaCO_3$. EDTA is sparingly soluble in water and hence disodium salts of EDTA is used as reagent for preparing the solution.

The structure 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 :

Name of Experiment: Determination of total hardness of water Date: 27 02 19
Experiment No: 01 Page No: 03

I Preparation of Standard solution of disodium salt of EDTA :

Weigh given disodium salt of EDTA accurately using an electronic balance. Transfer the crystals of EDTA into a clean 250 cm^3 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

II Determination of total hardness of Water :

Pipette out 25 cm^3 of the given hard water sample into a clean conical flask, to this add 5 cm^3 of ammonia-ammonium chloride buffer solution, and 2 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.

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Name of Experiment: Determination of total hardness of Water Date: 27 08 19

Experiment No. 01 Page No. 04

Reactions :

$M^{2+}(Ca^{2+}$ or $Mg^{2+}) + In \longrightarrow M\text{-}In$ (Wine red colour)

hard Water Indicator (Metal ion - In complex)

$M\text{-}In + Na_2H_2Y \longrightarrow M\text{-}Y + 2Na^+ + 2H^+ + In$

(blue) (Metal ion - EDTA complex)

Result

The total hardness of the given water sample is 634.608 ppm of $CaCO_3$

27/08/19

30/30

Observation and Calculations

1. Preparation of Standard solution of disodium salt of EDTA

Weight of weighing bottle + EDTA salt = 6.2785 g
Weight of empty weighing bottle = 4.0070 g
∴ Weight of EDTA salt taken = 2.2715 g

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

$$M_{EDTA} = \frac{2.2715}{372.24} \times 4 = 0.024408\ M$$

$$\therefore a = 0.024408\ M$$

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 : Erichrome black - T
End point : Wine red to clear blue colour.

Burette Reading (cm^3)	I	II	III
Final Readings	6.5	6.5	6.5
Initial Readings	0.0	0.0	0.0
Volume of EDTA run down	6.5	6.5	6.5

$\therefore$ Volume of EDTA required, V = 6.5 cm^3

Note : 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 = $\frac{(a) \times V \times 100}{1000}$

X = 0.01586 g of $CaCO_3$

$\therefore$ Weight of $CaCO_3$ present in 25cm^3 of hard water = X = 0.01586 g

∴ Weight of $CaCO_3$ present in $25cm^3$ of hard water in terms of ppm $= y = \frac{x \times 10^6}{25}$

$$y = \frac{0.01586 \times 10^6}{25}$$

$$y = 634.608 \text{ ppm}$$

Thus total hardness of water in terms of ppm =
$y = 634.608$ ppm

Note : Parts per million : ppm is the parts of $CaCO_3$ equivalent hardness present per million or 10^6 parts of water

ie : 500 ppm = 500 parts of $CaCO_3$ equivalent hardness present per million or 10^6 parts of Water.

Experiment no. : 02

Determination of calcium oxide in the given sample of cement solution by rapid EDTA method

Theory : Cement contains oxides of calcium, aluminium, 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 patter - Ruder's indicator. The latter is used in this titration because Erlochrome 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 from complexes with EDTA (i.e., Mg^{+2} ions do not react with EDTA in the above conditions)

Procedure

1. Preparation of Standard solution of Disodium salt of EDTA:

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

II Estimation of Calcium oxide:

Pipette out 25cm³ of the given cement solution into a clean conical flask. Add 5cm³ of 1:1 glycerol, 5cm³ of diethyl amine and 10cm³ of 4N sodium hydroxide. Add 3-4 drops of pattern Ruder's indicator and titrate this against EDTA solution taken in a burette until wine red colour changes to purple blue. Note down the volume of EDTA added and repeat the experiment for concordant values. Calculate the percentage of calcium oxide

Reactions

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

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

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

Result:

The amount of CaO present in the given solution is 0.484 g

03/09/19

30/30

Observation and calculations :

I. Preparation of standard solution of disodium salt of EDTA

Weight of weighing bottle + EDTA salt = 6.9870 g

Weight of empty weighing bottle = 4.6230 g

∴ Weight of EDTA salt taken = 2.364 g.

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

$$M_{EDTA} = \frac{2.364 \times 4}{372.24} = 0.02540\ M$$

II. Estimation of CaO in cement solution

Burette : Standard EDTA Solution

Conical flask : 25 cm^3 of cement solution + 5 cm^3 of 1:1 glycerol + 5 cm^3 of di-ethylamine + 10 cm^3 of 4N NaOH

Indicator : Pattern - Reader's indicator

End point : Wine red to purple blue colour

Burette reading	I	II	III
final reading	8.5	8.5	8.5
Initial reading	0.0	0.0	0.0
Volume of EDTA run down in cm^3	8.5	8.5	8.5

$\therefore$ Volume of EDTA required = V = 8.5 cm^3

Note : Molecular weight of CaO = 56.08

1000 cm^3 of 1M of EDTA = one gram molecular weight of CaO (56.08)

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

$$X = \frac{0.0254 \times 8.5 \times 56.08}{1000}$$

X = 0.0121076 g of CaO

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

X = 0.0121076 g

∴ Weight of CaO present in 1000 cm^3 of cement solution

$$y = \frac{x \times 1000}{25}$$

$$y = \frac{0.0121076 \times 1000}{25}$$

$$y = 0.484\ g$$

Result

Amount of CaO in the 1000 cm^3 of cement solution is 0.484g

Experiment no. : 03

Determination of percentage of copper in brass using standard sodium thiosulphate solution

Theory :

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

Cu = 50 - 90 %, Zn = 20 - 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 destroyed by boiling with urea. The solution is neutralized 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 indicator. The volume of sodium thiosulphate consumed is measure of the amount of copper present in

the solution and sodium thiosulphate which is commonly known as hypo.

Procedure :

I 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 untill the reddish brown fumes of oxides of nitrogen are expelled completely. Cool the solution to room temperature and transfer the solution of brass in to a clean 250 cm^3 standard flask. Make up the solution to the mark by adding distilled water and mix well for uniform concentration.

II. Estimation of copper

Pipette out 25 cm^3 of brass solution into a clean conical flask and add dilute ammonium hydroxide drop by drop untill 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 Iodide 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 ~~red~~ 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 :

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

$$2Cu(NO_3)_2 + 4KI \longrightarrow \underset{\text{Cuprous Iodide}}{Cu_2I_2\downarrow} + 4KNO_3 + I_2\uparrow \text{ (brown)}$$

$$\underset{\text{hypo}}{2Na_2S_2O_3} + I_2 \longrightarrow 2NaI + \underset{\text{Sodium tetra thionate}}{Na_2S_4O_6}$$

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

Result : The percentage of Cu present in the given brass sample is 77.05 %

17/9/19

30/30

Observations and calculations :

1. preparation of brass solution :

Weight of brass taken is = 0.4662 g (W)

II. Estimation of Copper :

Burette : Standard hypo (sodium thiosulphate) solution

Conical flask : 25 cm^3 brass solution + NH_4OH drop wise till a bluish white ppt. is formed. Dissolve the ppt. by adding dilute acetic acid + $1/4^{th}$ t.t of acetic acid in excess + one t.t of 10% KI + shake well + one t.t of distilled water

Indicator : 2 cm^3 of freshly prepared starch

End point : Disappearance of blue colour.

Burette reading	I	II	III
Final reading Initial reading	11.3 0.0	11.3 0.0	11.4 0.0
Volume of hypo run down in cm^3	11.3	11.3	11.4

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

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

∴ V cm^3 of 0.05 of hypo = $X = \frac{V \times 0.05 N \times 63.54}{1000} = \frac{11.3 \times 0.05 N \times 63.54}{1000}$

$X = 0.0359001$ g of copper

∴ Weight of copper present in $25 cm^3$ of brass solution = $X = 0.0359001$ g

∴ Weight of copper present in $250 cm^3$ of brass solution

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

$$Y = 0.359001 g$$

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

$$= \frac{0.359001 \times 100}{0.4662}$$

$y = 77.005\%$

$\therefore$ Percentage of copper in the given brass sample $= 77\%$

EXPERIMENT NO : 04

Determination of Iron in a given sample of Haematite ore solution.

Theory : Haematite is an important ore of iron containing mainly ferric acid (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 oxidized by adding mercuric chloric solution. The ferrous ions in the resulting solution is titrated against standard potassium dichromate using potassium ferricyanide as external indicator. Potassium ferricyanide produces an intense deep blue colour with ferrous ions due to the formation of ferriferrocyanide complex.

Name of Experiment

Date 24 09 19

Experiment No 04

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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 1/4 t.t of concentration hydrochloric acid and heat the solution nearly to boiling. add stannous chloride drop by drop taken in a separate burette till yellow colour solution becomes colourless 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 taken in a burette

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Experiment No. 4

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 hamatite 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)

$$3FeCl_2 + 2K_3[Fe(CN)_6] \longrightarrow 6KCl + Fe_3[Fe(CN)_6]_2 \text{ (deep blue)}$$

Potassium ferricyanide ferri ferro cyanide complex

Result :

The amount of iron in the given Haematite ore solution is 1.142 g

29/30

Observation and calculation

I. Preparation of Standard potassium dichromate Solution

Weight of weighing bottle + $K_2Cr_2O_7$ crystals = 5.0480 g
Weight of empty weighing bottle = 4.0425 g
∴ Weight of $K_2Cr_2O_7$ crystals taken = 1.0055 g

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

$$N_{K_2Cr_2O_7} = \frac{1.0055 \times 4}{49} = 0.08208 \text{ N (a)}$$

II. Estimation of Iron

Burette : Standard $K_2Cr_2O_7$ Solution

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

Indicator : potassium ferric cyanide as external indicator

End point : Failure to observe blue colour ; when a drop of reacting mixture is brough in contact with the indicator placed on paraffine paper

Burette readings	I	II	III
Final readings Initial readings	6.2 0.0	6.3 0.0	6.2 0.0
Volume of $K_2Cr_2O_7$ run down in cm^3s	6.2	6.3	6.2

$\therefore$ Volume of $K_2Cr_2O_7$ required $= V = 6.23\ cm^3$

Note : Equivalent weight of iron $= 55.85\ g$

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

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

$$X = \frac{(0.08208)(6.23)(55.85)}{1000} = 0.02855 \text{ gm of Iron}$$

$\therefore$ Weight of iron present in $25\ cm^3$ of haematite solution $X = 0.02855\ g$

$\therefore$ Weight of Iron present in $1000\ cm^3$ of Haematite ore solution $= y = \frac{X \times 1000}{25}$

$$y = \frac{0.02855 \times 1000}{25}$$

$$y = 1.142\ g$$

EXPERIMENT No : 05

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 oxygen 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 alcohol, 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

I **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 250 cm^3 standard flask. Dissolve the crystals by adding 100 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.

II **Determination of COD of Waste water**

Pipette out 25 cm^3 of Industrial waste water and add exactly 25 cm^3 of standard potassium dichromate solution (0.1N) into a clean conical flask. add one t.t of 1:1 sulphuric acid and 2 to 4 drops of ferroin indicator and titrate against standard Mohr's salt solution taken in a burette untill blue green colour changes to reddish-brown. Note down the volume of Mohr's salt solution added for unreacted potassium dichromate solution (V_1) and

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

III Blank titration

Add exactly 25 cm^3 of standard potassium dichromate solution into a clean conical flask and one tt 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 Waste water + Oxidising agent $\xrightarrow[H_2SO_4]{heat}$ $H_2O + CO_2\uparrow$

(Organic compounds) ($K_2Cr_2O_7$)

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

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ferric ion indicator complex $\xrightarrow{FAS}$ ferroin indicator (Fe^{2+})
(Fe^{3+}) $\xleftarrow{K_2Cr_2O_7}$

(blue green) (reddish brown)

Result

The COD of the given waste water sample contains 183.79 mg of Oxygen

01/10/19

29/30

Burette readings	I	II	III
Final readings Initial readings	8.2 0.0	8.0 0.0	8.2 0.0
Volume of FAS run down in cm^3	8.2	8.0	8.2

∴ Volume of FAS solution required = V_1 = 8.2 cm^3

IV. Blank titration of (FAS >< $K_2Cr_2O_7$)

Burette : Standard FAS solution
Conical flask : 25 cm^3 of $K_2Cr_2O_7$ solution + 1 lt of 1:1 H_2SO_4
Indicator : 4-6 drops of ferric indicator
End point : Blue green to reddish brown colour.

∴ Volume of FAS solution required for blank titration
V_2 = Final B.R − Initial BR = 14.5 − 0.0

V_2 = 14.5 cm^3

∴ Volume of $K_2Cr_2O_7$ required for the oxidation of organic compounds of waste water = $V_2 - V_1$
= 14.5 − 8.2
= 6.3 cm^3

1000 cm^3 of 1N of FAS = 1000 cm^3 of 1N $K_2Cr_2O_7$ solution = 8 g of O

$V_2 - V_1$ cm^3 of (a) N FAS = X = $\frac{8 \times (a) \times V_2 - V_1}{1000}$

$$X = \frac{8 \times 0.09116 \times 6.3}{1000}$$

$$X = 4.594 \times 10^{-3} \text{ g of Oxygen}$$

∴ Amounts of O_2 required to oxidise organic compounds present in 25 cm^3 of Waste water = X = 4.594×10^{-3} g

∴ Amount of O_2 required to oxidize organic compounds present in 1000 cm^3 of waste water = $Y = \frac{X \times 1000}{25}$

$$y = \frac{4.594}{25}$$

$$y = 0.1837 \text{ g}$$

∴ COD of Waste water = $Y \times 1000$ = 0.1837×1000 mg

∴ COD of Waste water = 183.79 mg of Oxygen

Experiment no. : 06

Determination of pKa value of a week acid using 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

$$HCOOH\,(aq) + H_2O\,(l) \xrightleftharpoons{Ka} H_3O^+\,(aq) + HCOO^-\,(aq)$$

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

06

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

pKa value is determined be measuring the changes in pH of acid solutions a different amount of the base added. In the titration of an acid with a base, 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 a 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 calullated using Henderson equation

$$pH = pKa + \log_{10} \frac{[Salt]}{[Acid]}$$

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

Procedure

Transfer given quantity of weak acid ($25 cm^3$) into a clean $100 cm^3$ 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 a beaker. Note down the pH of the acid. Add $0.5 cm^3$ of sodium hydroxide solution at a time taken in a micro burette in to the acid, stir the solution using magnetic stirrer and measure the pH of the acid and continue the addition of sodium hydroxide untill the solution sudden raise in the pH. Then take few more reading and plot the graph, pH against the volume of sodium hydroxide added. 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 and from the graph 2, determine the equivalent point of the acid. Determine the half equivalence point that gives the pKa value of weak acid by the graph 1

Name of Experiment

Experiment No 06

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Note : Switch off the ~~instruction~~ instrument after experiment is completed

Result

1. Equivalence point = 5.0
2. Half E.P = 2.5
3. pH at E.P = 10.1
4. pH at H.E.P = 5.2
5. pKa of the given acid = 5.2

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Observation and Calculation

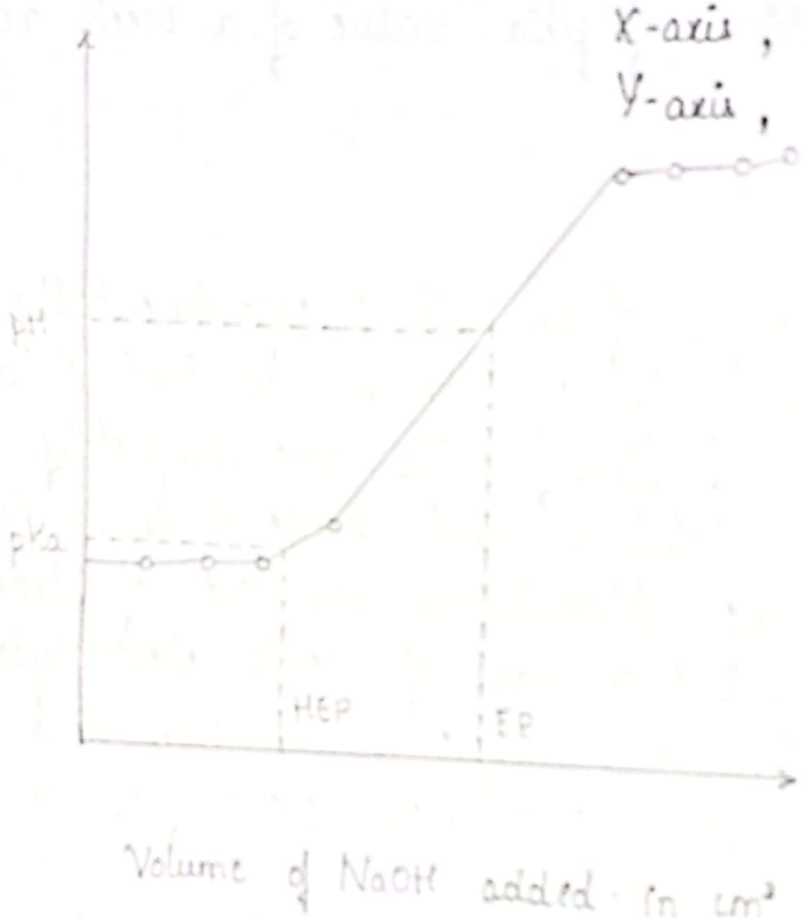
Volume of NaOH (cm^3)	pH	ΔpH	ΔV	ΔpH / ΔV
0.0	3.96	x		
0.5	4.43	0.47		0.94
1.0	4.69	0.26		0.52
1.5	4.89	0.20		0.4
2.0	5.05	0.16		0.32
2.5	5.23	0.18		0.36
3.0	5.38	0.15	05	0.3
3.5	5.60	0.37		0.74
4.0	5.93	0.33		0.66
4.5	6.90	0.97		1.94
5.0	10.13	3.23		6.46
5.5	10.72	0.59		1.18
6.0	10.93	0.21		0.42

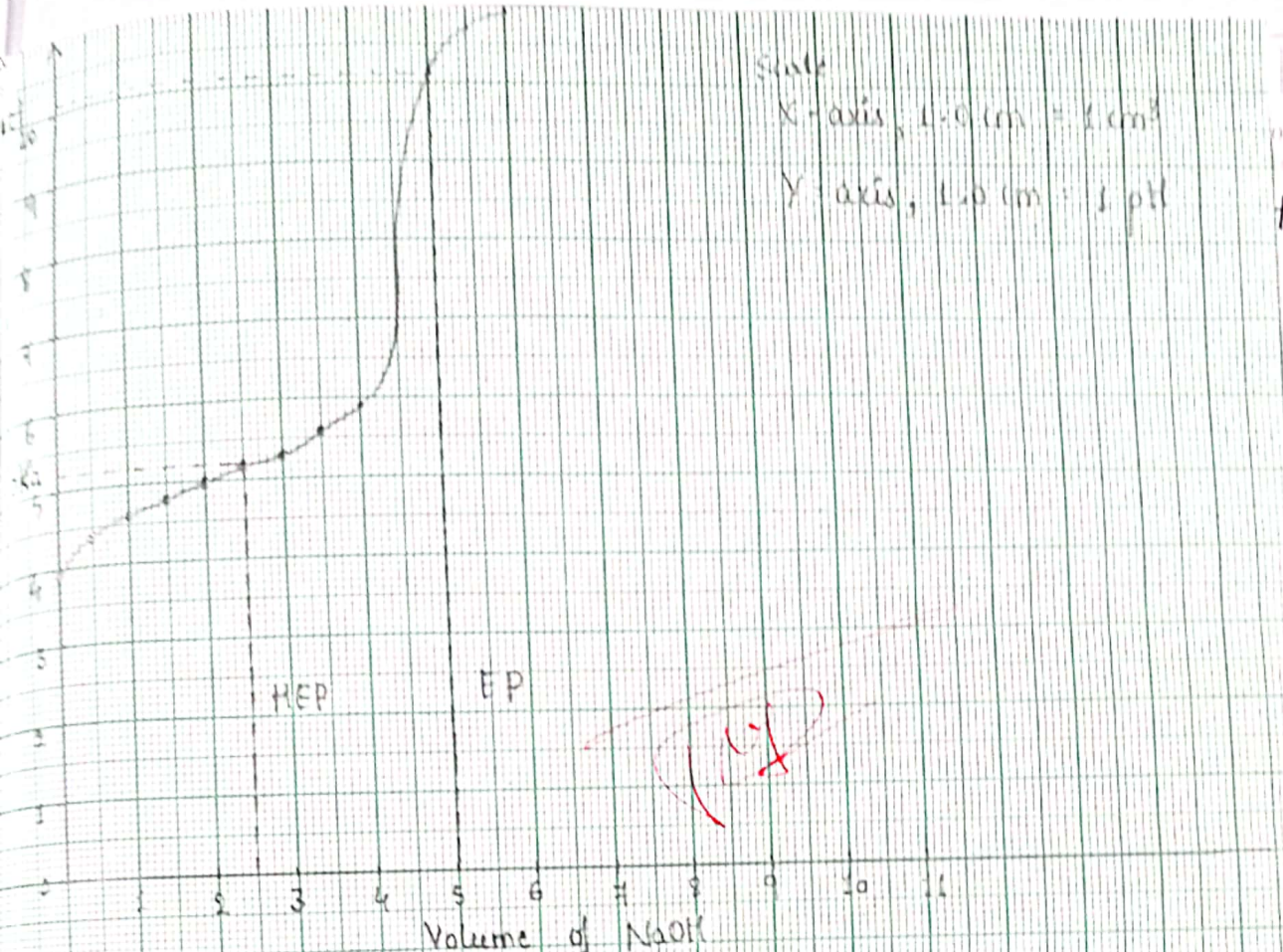
Result

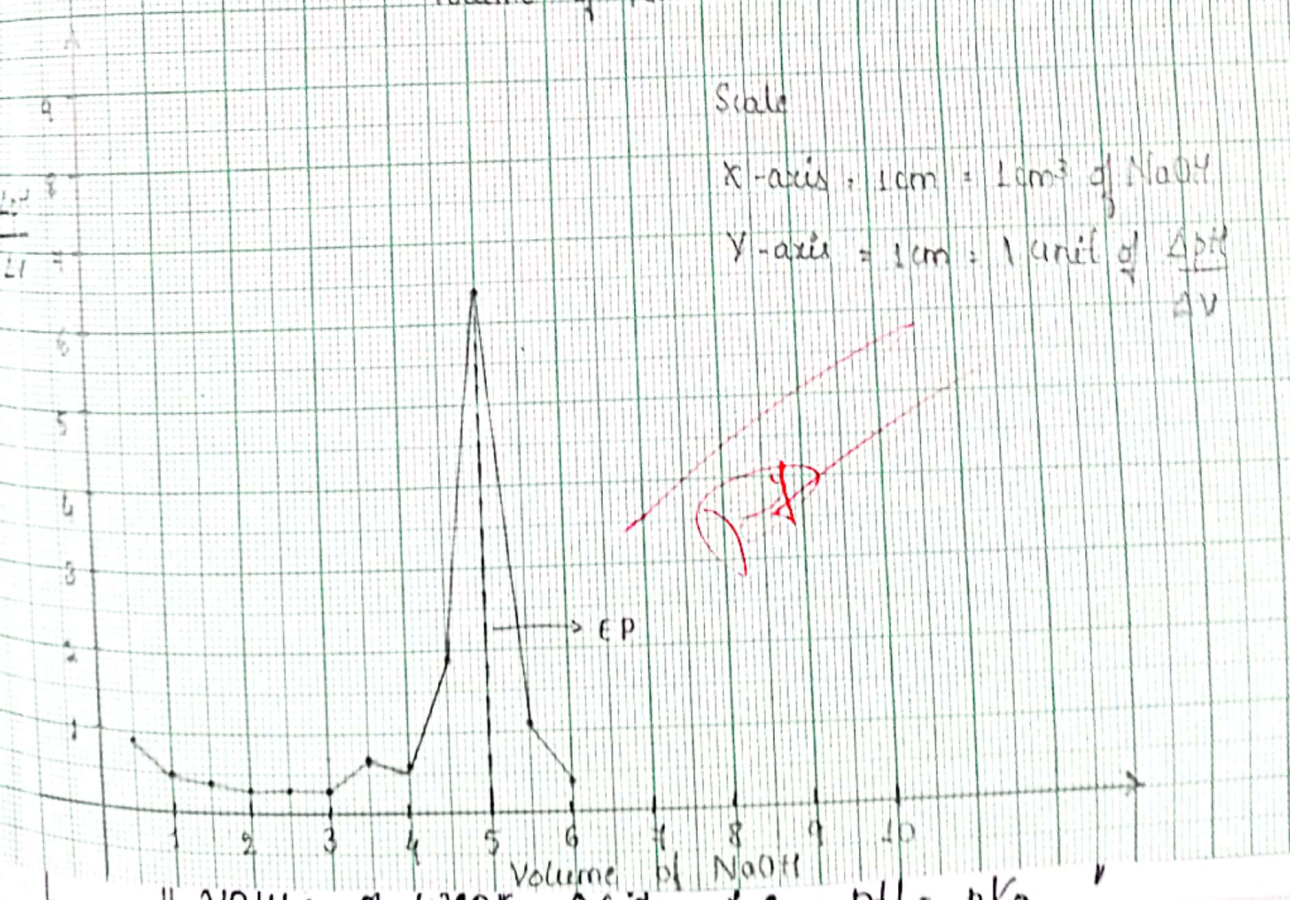
1. Equivalent point = 5.0
2. Half E.P = 2.5
3. pH of E.P = 10.1

4. pH of HEP = 5.2
5. pKa of a given acid = 5.2

Scale
X-axis, 1.0 cm = 1 cm³
Y-axis, 1.0 cm = 1 pH







value of weak acid i.e. pH = pKa

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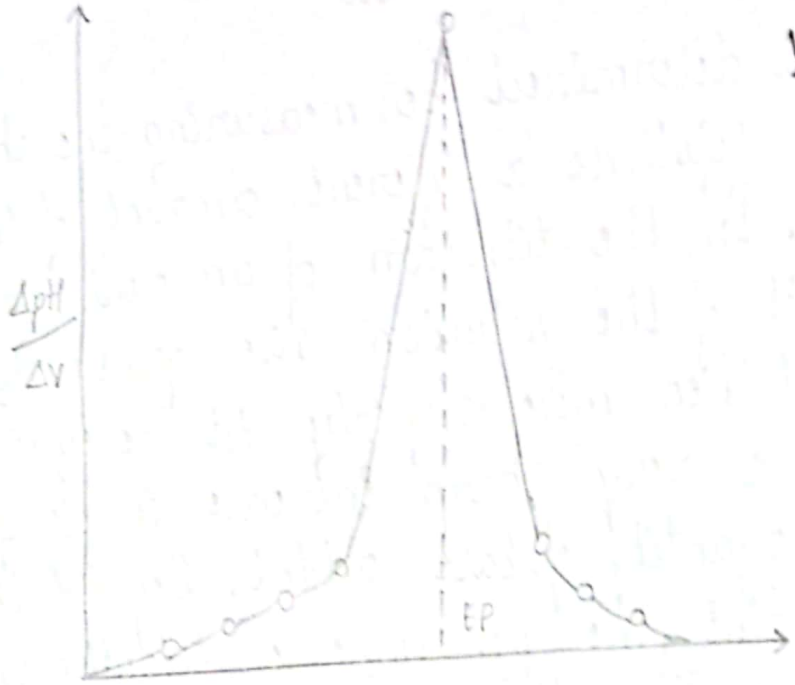
Scale

X-axis , 1.0 cm = 1 cm

Y-axis , 1.0 cm = 1 unit



Volume of NaOH added in cm^3

Experiment no : 07

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 molecules layers. A layer close to the surface is almost stationary while that at the axis of tube move 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 coefficient of viscosity is defined as the tangential force per unit area required to maintain a unit velocity gradiant between the any two successive layers of liquid situated unit distance apart. The coefficient of viscosity of a liquid is given by poiseuille's formula,

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

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

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, g = gravitational force. If equal volumes of two different liquids are allowed to flow through the same tube under identical condition, 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 and η_L = co-efficient of viscosity of the given liquid which can be determined. t_w = time taken by the standard liquid, t_L = time taken by the given liquid, d_w = density of standard liquid, d_L = density of given liquid. Viscosity is a temperature dependent property and is inversely proportional to the temperature (T) i.e $\eta \propto 1/T$. Hence the measurments are carried out of 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 the burette into the viscometer. suck the distilled water above the upper mark A in the viscometer. ~~suck 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 of the given organic liquid into a viscometer. Note down the time of flow of organic liquid 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

Name of Experiment

Experiment No. 07

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Result :

$\therefore \eta_L$ = Co-efficient of Viscosity of liquid = 29.868 milli poise

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Observation and calculation

Trial no.	I	II	III	Average time in sec
Time taken by water in sec (t_w)	63	63	63	63
Time taken by liquid in sec (t_L)	221	225	232	226

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

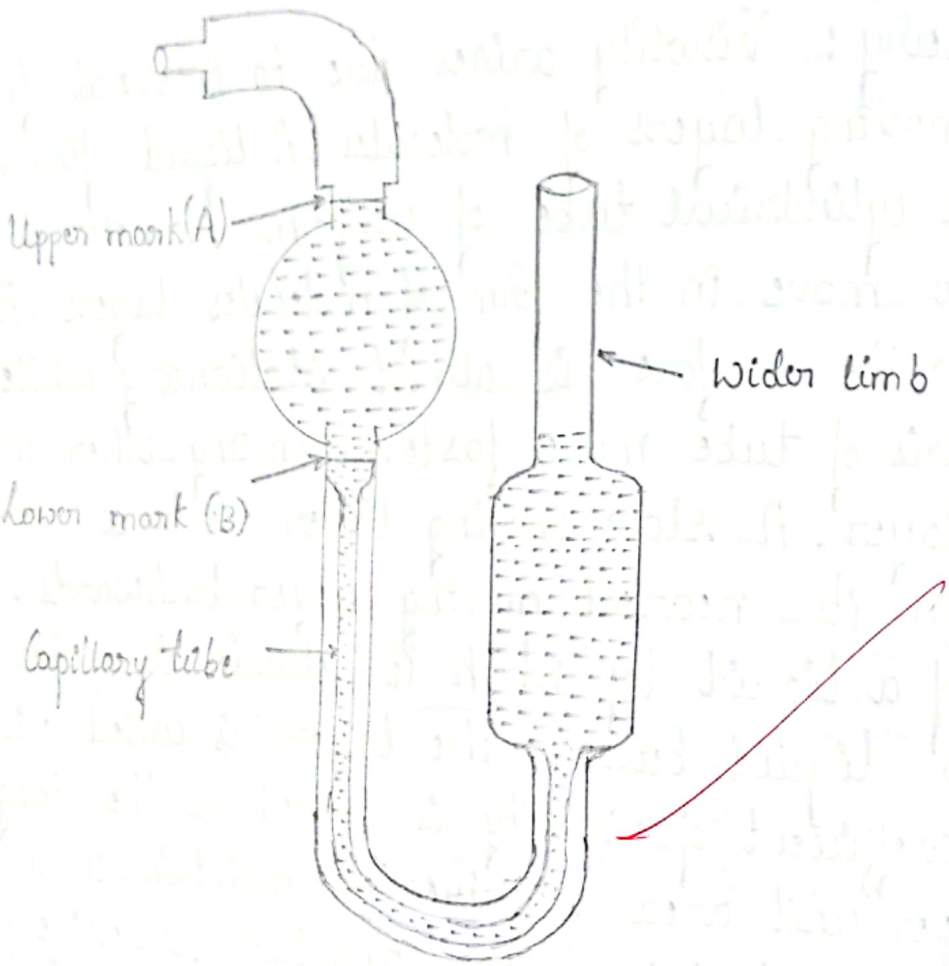
where t_w = time taken by water = 63 sec
t_L = time taken by liquid = 226 sec
d_w = density of water = 0.9959 g/cc
d_L = density of liquid = 1.0137 g/cc

η_w = co-efficient of viscosity of water = 8.18 mille poise

η_L = co-efficient of viscosity of liquid = 28.86 milli poise

$$\eta_l = \frac{\eta_w t_l d_l}{t_w d_w} = \frac{8.18 \times 226 \times 1.0137}{63 \times 0.9959} \text{ milli poise}$$

$\therefore \eta_l$ = co-efficient of viscosity of liquid = 29.868 milli poise



VISCOMETER

Experiment no : 08

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 the electrolyte, an electro chemistry 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 measured. 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,

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

where :

E_{cell} = emf of the cell, E°_{cell} standard emf of the cell,
n = number of electrodes involving in the reaction,
[Products] = concentration of the products and
[Reactants] = concentration of the reactants.

Procedure

Transfer the given amount of Mohr's salt solution in to a clean 100 cm^3 beaker. to that add one test tube of dilute sulphuric acid.

Switch on the potentiometer and adjust the temperature knob to the lab temperature. connect the platinum and calomel electrodes to the potentiometer. Wash the electrodes with ~~dtl~~ distilled water and immerse the electrodes in to the Mohr's salt solution taken in the beaker. Measure the emf of the solution in terms of mV. Add 0.5 cm^3 of potassium dichromate solution at a time taken in a micro burette into the Mohr' salt solution. Stir the solution at a time taken in a micro burette into the Mohr's salt

solution. Stir the solution and measure the emf of the solution and continue the addition potassium dichromate added untill the solution raise in the 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, 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 7.056 g

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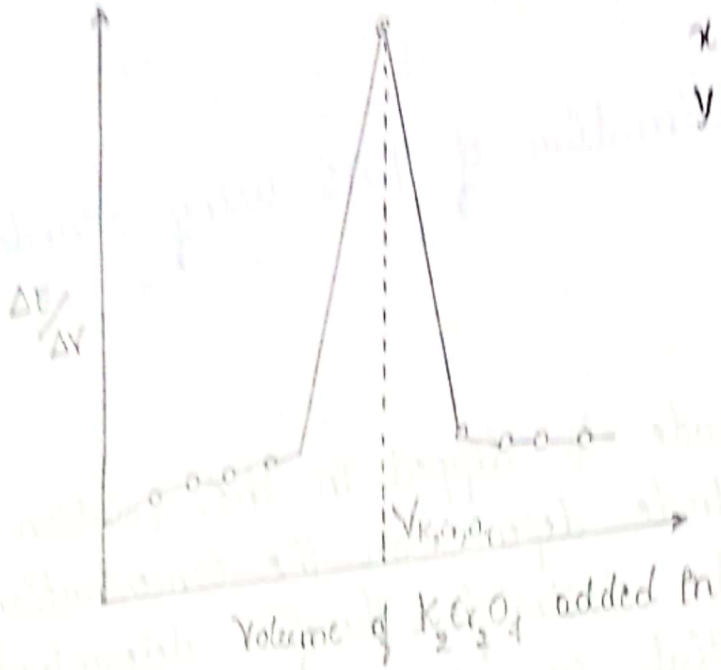
Volume of $K_2Cr_2O_7$	EMF (E) in mV	ΔE	ΔV	$\Delta E/\Delta V$
0.0	423	**		
0.5	438	15		30
1.0	449	11		22
1.5	459	10		20
2.0	469	10		20
2.5	479	10	0.5	20
3.0	492	13		26
3.5	507	15		30
4.0	536	29		58
4.5	835	299		598
5.0	848	13		26
5.5	865	17		34

Scale

x axis, 1.0 cm = 1 cm^3

y axis, 1.0 cm = 50 units



$\therefore$ Volume of $K_2Cr_2O_7$ required : $V_{K_2Cr_2O_7}$ = 4.5 cm^3

Note :

1. Normality of $K_2Cr_2O_7$ = $N_{K_2Cr_2O_7}$ = 0.1 N

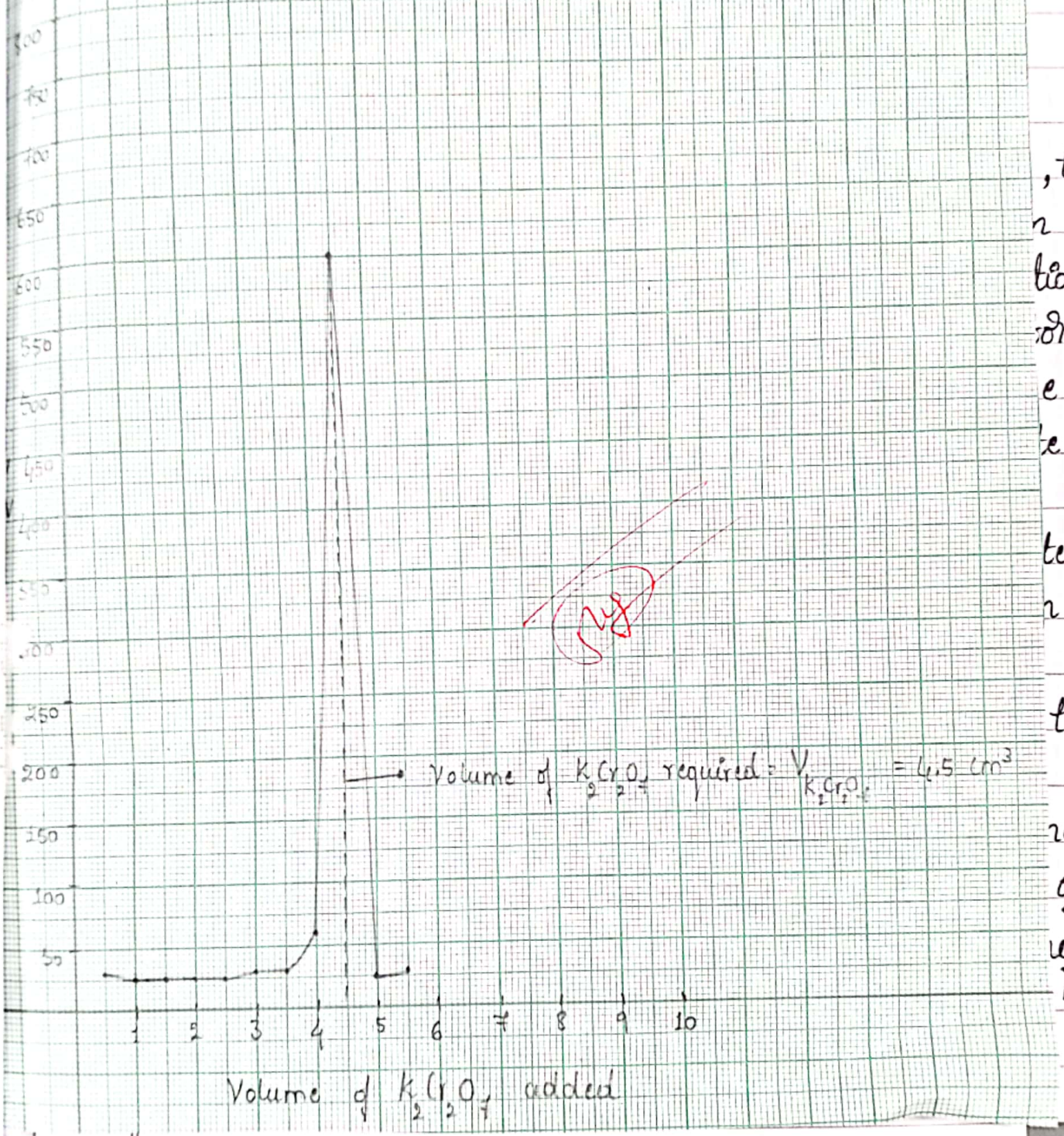
2. Equivalent weight of Mohr's salt = 392

$\therefore$ Normality of Mohr's salt solution = $\dfrac{N_{K_2Cr_2O_7} \times V_{K_2Cr_2O_7}}{V \text{ Mohr's sal}}$

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

X axis, 1.0 cm = 1 cm^3

Y-axis, 1.0 cm = 50 units



Weight per liter of Mohr's salt crystals $= X = N_{\text{Mohr's salt}} \times$ equivalent weight of Mohr's salt

$X = 0.018 \times 392 = 7.056\ g$

∴ Amount of Mohr's salt present in the given solution is $= 7.056\ g$

Result

Amount of Mohr's salt present in the given solution is $7.056\ g$

Name of Experiment
Experiment No. 09

Experiment no. : 09

Conductometer estimation of acid mixture using standard sodium hydroxide

Theory

Measurment of conductance of 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 graphically by plotting conductance (ordinate) against sodium hydroxide (abscissa)

Conductometric titration may be applied for the determination of acid and acids present in a mixture. In the titration of a mixture of acid with a strong base (sodium hydroxide) the conductance falls untill 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 (25 cm^3) into a clean 100 cm^3 beaker. Fill standard NaOH (0.2 N) solution into 10 cm^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 0.5 cm^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 ~~di~~ titration proceeds the conductance is gradully a decreases and then increases slowly and finally increased sharply. Take 5-6 readings when conductance begins to increases 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 liter of HCl and CH_3COOH

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

Result

a) The weight of HCl / dm^3 0.7884 g

b) The weight of CH_3COOH / dm^3 2.06572 g

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$NaOH + HCl \longrightarrow$

$NaOH + CH_3COOH \longrightarrow CH_3COONa + H_2O$

Observations and calculation

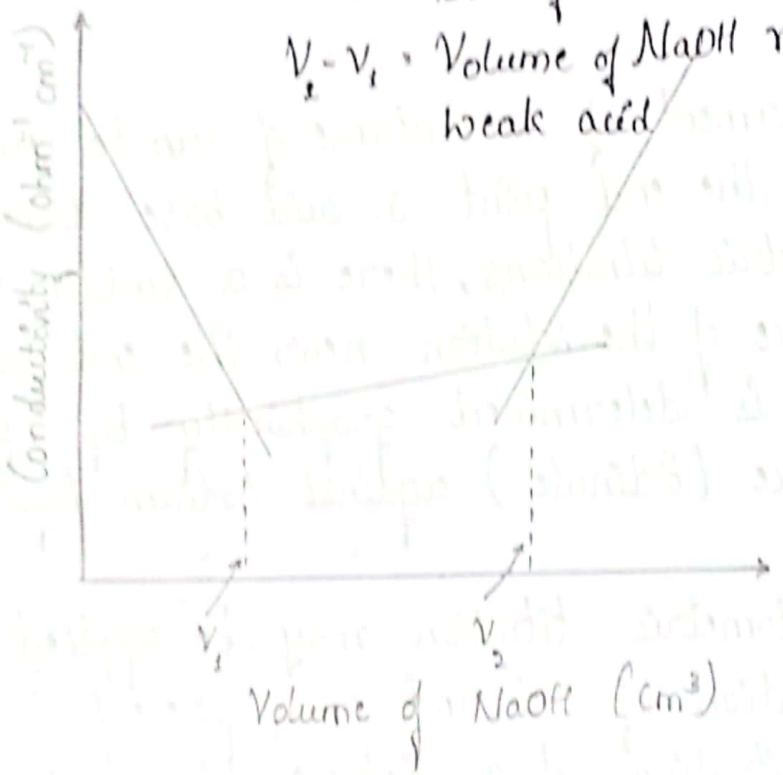
Estimation of acid mixture

Volume of NaOH in cm^3	Conductance $Ohm^{-1} cm^{-1}$
0.0	5.97
0.5	5.21
1.0	4.40
1.5	3.63
2.0	2.82
2.5	2.09
3.0	1.99
3.5	2.14
4.0	2.29
4.5	2.47
5.0	2.63
5.5	2.78
6.0	2.94
6.5	3.09
7.0	3.27
7.5	3.70
8.0	4.15
8.5	4.57
9.0	4.96

Volume of NaOH in cm^3	Conductance ohm^{-1} cm^{-1}
9.5	5.29
10.0	5.66

V_1 = Volume of NaOH required to neutralize strong acid

$V_2 - V_1$ = Volume of NaOH required to neutralize weak acid



Estimation of HCl

Volume of NaOH required to neutralize HCl = V_1 = 2.7 cm^3

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

$$= \frac{0.2 \times 2.7}{25} = 0.0216 \text{ N}$$

Weight per litre of HCl = X = N_{HCl} X Equivalent weight of HCl

$$X = 0.0216 \times 36.5$$

$$X = 0.7884 \text{ g}$$

Estimation of CH_3COOH

Volume of NaOH required to neutralize CH_3COOH =

$$(V_2 - V_1) = 4.3 \text{ cm}^3$$

Normality of CH_3COOH = $\frac{N_{NaOH} \times V_{(V_2-V_1)}}{25}$ = $\frac{0.2 \times 4.3}{25}$

$$= 0.0344 \text{ N}$$

Weight per litre of CH_3COOH = X = N_{CH_3COOH} X Equivalent weight of CH_3COOH

$$X = 0.0344 \times 60.05$$

$$X = 2.06572 \text{ g}$$

EXPERIMENT NO : 10

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 ∝ 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 620 nm since the complex shows maximum absorbance of 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 25 cm^3 of the solution into 50 cm^3 standard flasks. Add 5 cm^3 of 1:1 ammonia solution to each of them and also to the test solution given in a separate 50 cm^3 standard flask

Make up the mark solution to the mark by adding distilled water and mix well for uniform concentration prepare a blank solution by adding 5 cm^3 of 1 : 1 ammonia solution into a 50 cm^3 standard flask make up the solution to the mark with distilled water and mix well for uniform concentration

Switch on the colorimeter and warm it for 5 minutes. Using the filter knob (B), select the wave length of λ_{max} = 620 nm for copper sulphate solution. Take the blank solution in the cell tube and keep it in the calorimeter and adjust zero absorbance for 100% transmittance using the knob(A) Then measure the optical density or absorbance for each standard solution and test solution using a photo electric colorimeter. A straight line graph is obtained by plotting optical density or absorbance against the concentration of copper. From the graph determine the concentration of copper in the test solution and calculate the volume of copper sulphate in the given test solution.

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

Cuprammonium sulphate

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

1 The Volume of test solution given = 9.5 cm^3
2 Absorbance of test solution = 0.10
3 Concentration of copper in test solution = 9.5 mg/cm^3
4 Concentration of copper sulphate solution = 37.3 g/cm^3

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Observations and calulations

Volume of copper sulphate in cm^3	Concentration of copper sulphate in mg / cm^3	Concentration of copper in mg / cm^3	Absorbance or OD at 620nm
5	3.93 x 5 = 19.65	0.5 x 1 = 0.5	0.05
10	3.93 x 10 = 39.30	10 x 1 = 10	0.10
15	3.93 x 15 = 58.95	15 x 1 = 15	0.15
20	3.93 x 20 = 78.60	20 x 1 = 20	0.21
25	3.93 x 25 = 98.25	25 x 1 = 25	0.25
Test Solution	3.93 x 9.5 = 37.3	10 x 1 = 10	0.10

Note : Molecular weight of copper sulphate pentahydrate = 249.54

i) Atomic weight of copper = 63.54.

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

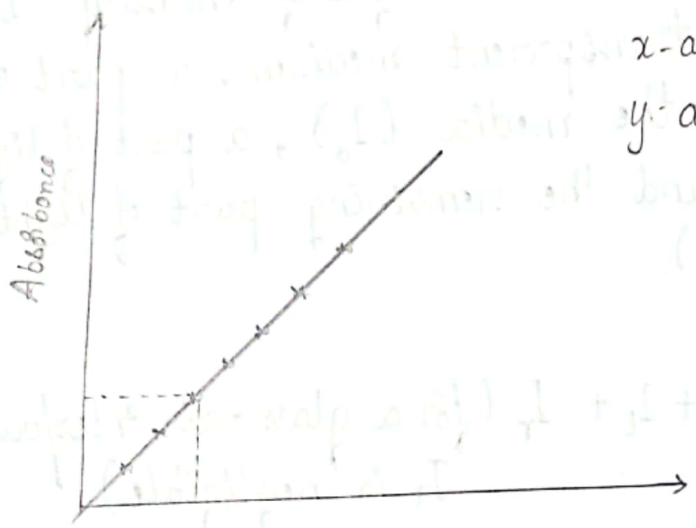
249.54 g of $CuSO_4 \cdot 5H_2O$ = 63.54 g of copper

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

Scale

x-axis = 1.0 cm = 5 mg/cm³

y-axis = 1.0 cm = 0.05 OD



Concentration of Cu in mg/cm³

Date 19/11/19

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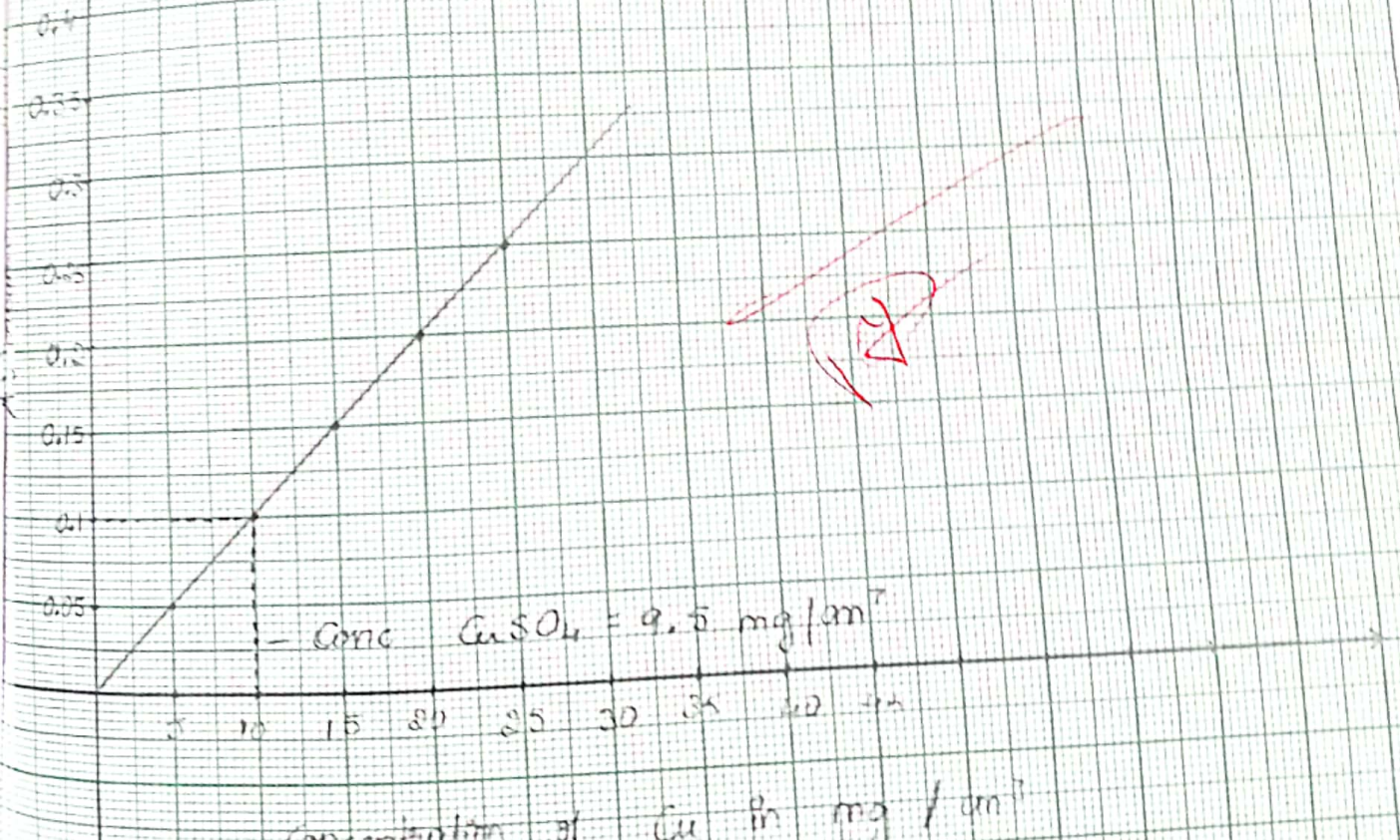
Scale

x axis - 1.0cm - 5 mg/cm³

y axis - 1.0cm - 0.500



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$\therefore$ 1.0 cm^3 of $CuSO_4$ = 3.93 mg of $CuSO_4$ = 1.0 mg of copper

C = Concentration of copper in test solution = 9.5 mg / cm^3

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

$\therefore$ V cm^3 of copper sulphate = $V = \frac{C}{10} = \frac{9.5}{1.0}$

$= \underline{9.5}\ cm^3$

Hence, the volume of test solution given = V = 9.5 cm^3