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K. S. INSTITUTE OF TECHNOLOGY

KAMMAVARI SANGHAM (R) - 1952

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Laboratory Certificate

This is to certify that Mr./Ms. J NANESH. A.S
has satisfactorily completed the course of experiments in
Engineering chemistry..... laboratory, Code 18CHE26
prescribed by Visvesvaraya Technological University, Belagum for the
II..... Semester B.E..... CSE..... Branch
in this college during the academic year 20.20... - 20.21...

Name of the Candidate : J NANESH. A.S

USN : 1K.S.20CS043..... Subject (with code) Engineering chemistry (18CHE26)

Internal assessment marks awarded :

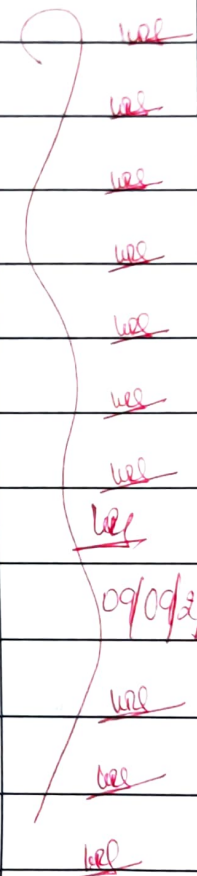
<u>30</u>	<u>09</u>	<u>39</u>
<u>30</u>	<u>10</u>	<u>40</u>

Signature of Staff Incharge

Date : 09/09/21

Signature of Head of
Department

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12	06/09/21	09/09/21	30	Flame photometry	20	10	30	

observation and calculation

Part A:

weight of the bottle + Mohr's salt = $W_1 = 25.75\text{g}$

weight of the empty bottle = $W_2 = 18.198\text{g}$

weight of the Mohr's salt = $(W_1 - W_2) = 7.552\text{g}$

$$N_{FAS} = \frac{(W_1 - W_2) \times 4}{\text{Eq. wt of FAS (392)}}$$

$$= \frac{7.552 \times 4}{392}$$

$$N_{FAS} = 0.077\text{ N}$$

Part B:

Sample titration

Burette: Standard FAS

Conical flask: 25.00 ml of waste water sample + 10.0 ml of $\text{K}_2\text{Cr}_2\text{O}_7$ + 10 ml of 1:1 H_2SO_4

Indicator: 2-3 drops of ferroin indicator

colour change: Bluish green to reddish brown.

DATE 1/09/21

EXPT TITLE:

EXP NO. 01

PAGE NO. 01

Determination of COD in waste water.

Aim: To estimate the chemical oxygen demand in waste water.

Principle: Chemical oxygen demand parameter has been introduced to measure total oxidisable impurities present in the sewage. This includes both biologically oxidisable and biologically inert but chemically oxidisable impurities in the water. It is the amount of oxygen equivalent of acidified potassium dichromate required to oxidize the organic and inorganic impurities in waste water. It is expressed in $\text{mg} \cdot \text{dm}^{-3}$ or ppm. A known volume of waste water sample is refluxed with excess of $\text{K}_2\text{Cr}_2\text{O}_7$ solution in sulphuric acid medium and in the presence of Ag_2SO_4 and HgSO_4 . $\text{K}_2\text{Cr}_2\text{O}_7$ oxidizes all oxidisable impurities. Ag_2SO_4 catalyses the oxidation of straight chain organic compounds, aromatics and pyridine. HgSO_4 avoids the interference of Cl^- ion by forming soluble complex with them. In the absence of HgSO_4 , Cl^- ions precipitate silver ions as AgCl . The amount of unreacted $\text{K}_2\text{Cr}_2\text{O}_7$ is determined by titration with standard Mohr's salt solution. The amount of $\text{K}_2\text{Cr}_2\text{O}_7$ solution consumed corresponds to the COD of the water sample. To calculate the COD, a blank titration is carried out.

Ferroin [(1,10-phenanthroline-iron (II)) complex] is used as the indicator in the oxidized form, imparting bluish green end point. The indicator is intensely red in colour. Strong oxidizing agents oxidize the indicator to iron (III) complex, which has a bluish green colour.

Burette reading in ml	I	II	III
Final burette reading	5.9	5.9	6.0
Initial burette reading	0	0	0
Volume of FAS consumed	5.9	5.9	6.0

Concordant value $V_1 = 5.9$ ml

Blank titration:

Burette : Standard FAS

Conical flask : 10.0 ml of Standard $K_2Cr_2O_7$ + 1:1 Sulphuric acid

Indicator : Ferroin

Colour change : Bluish green to reddish brown

Burette reading in ml	I
Final burette reading	35.0
Initial burette reading	0
Volume of FAS consumed	35.0

DATE

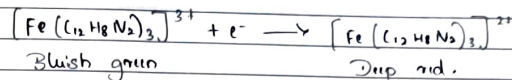
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EXP NO

01

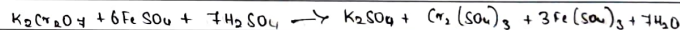
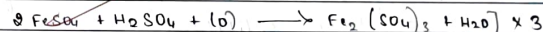
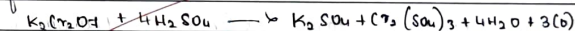
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As long as $K_2Cr_2O_7$ solution is present in the solution the indicator is in the oxidized form, imparting bluish green colour to the solution. The solution becomes bluish green as the titration proceeds and when $K_2Cr_2O_7$ is completely exhausted, the added FAS solution which is in oxidizing agents impart red-brown colour to the solution. Therefore, the endpoint is marked by the colour change from bluish green to reddish brown. Addition of con. H_2SO_4 to $K_2Cr_2O_7$ facilitates the liberation of nascent oxygen required for oxidation of waste water. A test tube of dil. H_2SO_4 is added during dissolution of Mohr's salt (Standard solution) to prevent the hydrolysis of the salt in the solution.

The reaction b/w Mohr's salt and $K_2Cr_2O_7$ can be represented as follows.



$$(NV)_{FAS} = (NV)_{\text{oxygen for waste water}}$$

$$N_{\text{oxygen for waste water}} = \frac{N_{FAS} V_{FAS}}{V_{\text{waste water}}} \quad (V_{FAS} = V_{\text{blank titrate value}} - V_{\text{sample titrate value}})$$

$$= \frac{0.077 \times 29.1}{25}$$

$$= 0.0896 \text{ N}$$

$$\text{Wt of oxygen / litre of waste water} = N_{\text{oxygen for waste water}} \times \text{Eq. wt of oxygen (8)}$$

$$= 0.0896 \times 8$$

$$= 0.7168 \text{ g [say]}$$

$$\text{Wt of oxygen in mg/ltr of waste water} = a \times 1000$$

$$= 0.7168 \times 1000$$

$$= 716.8 \text{ mg/ltr.}$$

DATE

EXPT. TITLE:

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01

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03

Procedure:Part A: Preparation of standard Mohr's salt solution:

Weigh accurately the given Mohr's salt, transfer it into a 250ml standard flask, dissolve in a little amount of distilled water and a little of dilute H_2SO_4 and make up to the mark with distilled water. Shake well for uniform concentration.

Part B: Determination of COD:

Pipette out 25.0ml of the waste water sample into a 250ml conical flask. Pipette out 10.0ml of 0.1N $\text{K}_2\text{Cr}_2\text{O}_7$ soln into the flask and add 10ml of 1:1 H_2SO_4 with constant shaking of the flask. Add 2-3 drops of Feroin indicator and titrate against standard Mohr's salt solution till the colour changes from bluish green to reddish brown.

Blank Titration:

Repeat the above procedure by taking 25ml-distilled water in the place of waste water sample.

Result: COD of the given sample of water is:

716.8 mg/dm³

Record

10

observation

10

viva

10

Total

30

Date: 09/09/21

KSIT

Observation and calculation

Vol of brass solution = 9 ml

Burette : standard sodium thiosulphate solution

Conical flask: 25 ml of brass solution + NH_4OH (drop wise till bluish white ppt) + CH_3COOH (to dissolve the ppt and 2-3 drops excess) + 10 ml of KI

Indicator : starch (near the end point)

colour change: Deep blue to milky white

Volume of Brass sol. (ml)			
Burette reading in ml	I	II	III
Final burette reading	10.6	11.7	11.7
Initial burette reading	0.0	0.0	0.0
Volume of $\text{Na}_2\text{S}_2\text{O}_3$	10.6	11.7	11.7

Any of the appropriate titre value (say 6 ml) is to be considered for calculations.

DATE 1/09/21

EXPT. TITLE:

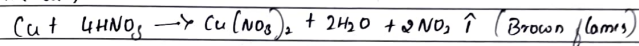
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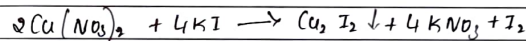
Estimation of Copper in Brass

Aim: To estimate the percentage of copper in given sample of brass.

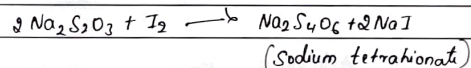
Principle: Brass is an alloy of copper and zinc. When dissolved in conc. nitric acid, both the metal get converted into their nitrates.



When brass solution is treated with excess of potassium iodide solution, cupric ions oxidize potassium iodide to liberate equivalent quantity of iodine. During the liberation of I_2 from KI, Cu^{2+} gets reduced to Cu^+ with a change in oxidation state by 1.



The liberated iodine is treated with sodium thiosulphate solution using starch as indicator.



The oxides of nitrogen present in the brass solution are destroyed by adding conc. The presence of oxides of nitrogen if not destroyed will be responsible for the liberation of extra iodine from KI, as they are also good oxidizing agents, thus increasing the titre value.

Normality of sodium thiosulphate used = 0.02 N (Say a)

100 ml of 1N $\text{Na}_2\text{S}_2\text{O}_3$ solution = 63.54 g of Cu

6 ml of a x N $\text{Na}_2\text{S}_2\text{O}_3$ solution = $\frac{6 \times a \times 63.54}{1000}$ g of Cu

$$= \frac{11.7 \times 0.02 \times 63.54}{1000} = 0.0148 \text{ (Say c)}$$

$$\% \text{ of Cu} = \frac{C \times 100}{W} \quad (W = 1g)$$

$$= \frac{0.0148 \times 100}{1}$$

$$= 1.48\%$$

DATE

EXPT. TITLE :

EXP. NO. 02

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The excess of nitric acid present in the brass solution is neutralised by adding NaOH solution till a pale blue precipitate of $\text{Cu}(\text{OH})_2$ is obtained. Other mineral acids are not preferable, as they will bring down the pH of a very small value, at which the liberation of I_2 from KI by Cu^{2+} precipitate is already is not quantitative and pH of experiment is hence maintained at around 5.

As it is titrated with $\text{Na}_2\text{S}_2\text{O}_3$, free Iodine reacts with it. Near the end point, when almost free iodine is exhausted in the solution, starch is added. Starch react with little iodine to form a blue coloured complex. Further addition of sodium thiosulphate dissociates the starch-iodine complex, consumes iodine and liberates starch, thereby discharging the blue colour.

Starch indicator is added near the end point and not in the beginning for the following reasons. (i) at high concentration of Iodine, starch may form an insoluble starch- I_2 complex.

(ii) The iodine also gets absorbed on colloidal starch particles, in the starch solution. The absorbed iodine cannot be liberated by the addition of sodium thiosulphate, and amount of iodine getting adsorbed is more when iodine concentration is high. Thus the error is minimized by adding starch near the end point.

Procedure:

Given amount of brass solution taken in 250. ml standard flask and make upto the mark with distilled water. pipette out 25. ml of brass solution in conical flask, add NH_4OH drop wise until a bluish white precipitate persists.

Add acetic acid in excess drop wise till bluish white precipitate disappears then add 2 drops of acid in excess, followed by 10 ml of 10% KI. Titrate the liberated iodine against sodium thiosulphate solution taken in the burette until the colour of the solution changes to pale yellow. Then add starch solution till solution becomes blue in colour, and continue the titration till the blue colour changes to milky white. Repeat the titration for concordant values.

Result: Percentage of copper in the given sample of brass is.
 $= 1.48\%$

Record :	10
observation :	10
viva :	10
Total :	30

09/09/21

observation and calculations:

part A:

Wt of the bottle + $K_2Cr_2O_7$ crystals = $W_1 = 20.678g$

Wt of the empty bottle = $W_2 = 17.121g$

Wt of $K_2Cr_2O_7$ crystals = $(W_1 - W_2) = 3.55g$

$$N_{K_2Cr_2O_7} = \frac{(W_1 - W_2) \times 4}{Eq. wt of K_2Cr_2O_7 (49)} = \frac{3.55 \times 4}{49}$$

$$= 0.289 N$$

Part B:

Burette: standard $K_2Cr_2O_7$

Conical flask: 5ml of haematite ore soln + 5ml of 4N HCl + boiled $SnCl_2$ drop wise till soln becomes colourless, 5 drops in excess + cool + 5ml of $HgCl_2$ + 2tt of water

Indicator: Potassium ferricyanide (external)

Colour change: disappearance of blue colour.

Burette reading in ml	I	II	III
Final burette reading	4.5	3.9	4.0
Initial burette reading	0	0	0
Volume of $K_2Cr_2O_7$	4.5	3.9	4.0

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EXPT. TITLE:

EXP. NO. 03

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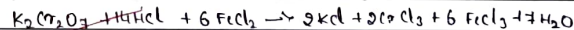
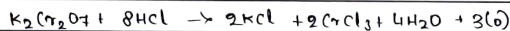
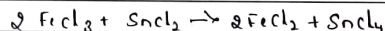
Estimation of Iron in Haematite

Aim: To estimate the percentage of iron in haematite ore solution.

Principle: Haematite is an oxide ore of iron, existing as ferric oxide (Fe_2O_3). On treating with HCl, ferric oxide dissolves in the acid as ferric chloride.



Since iron in $FeCl_3$ is already in the highest possible stable oxidation (Fe^{3+}), it cannot be oxidized further by potassium dichromate solution, which is also an oxidizing agent. Therefore, it is first reduced to ferrous state and then titrated with potassium dichromate. Ferric chloride is reduced by stannous chloride in presence of hydrochloric acid.



From the eqn, 2 moles of $FeCl_2 = 2$ moles of $FeCl_3 = 1$ atom of oxygen

A slight excess of stannous chloride is added to ensure complete reduction of $FeCl_3$ to $FeCl_2$. The excess of $SnCl_2$ added is good destroyed by adding strong solution of mercuric

Wt of the haematite ore given in 250 ml = $W_g = 4.125g$

$(NV)_{K_2Cr_2O_7} = (NV)_{Fe}$ in haematite ore

$$N_{Fe} = \frac{(NV)_{K_2Cr_2O_7}}{V_{Fe}} = \frac{0.289 \times 4.0}{25}$$

$$N_{Fe} = 0.0464 N$$

Wt of Fe/ltr of ore = $N_{Fe} \times \text{atomic weight of Fe (55.85)}$

$$= 0.0464 \times 55.85 = 2.5919 \text{ (say a)}$$

$$\text{Wt of Fe / 250 ml of ore} = \frac{a}{4} = \frac{2.591}{4}$$

$$= 0.649 \text{ g (say b)}$$

$$\text{percentage of Haematite} = \frac{b \times 100}{W}$$

$$= \frac{0.649 \times 100}{4.125}$$

$$= 15.73\%$$

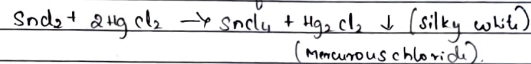
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EXPT. TITLE :

EXP. NO. 03

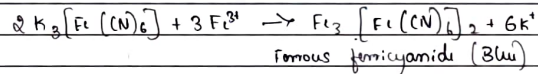
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chloride. $SnCl_2$ is a good reducing agent and can react with potassium dichromate if excess $SnCl_2$ is not neutralized.



A black precipitate of finely divided mercury may be produced if too much $SnCl_2$ is present. The finely divided mercury reduces $K_2Cr_2O_7$. Therefore the trial must be discarded if a black precipitate is produced. The trial should also be discarded if no precipitate is obtained on addition of mercurous chloride because the complete reduction of ferric ions of ferrous ions is not ensured.

Potassium ferricyanide is used as an external indicator to detect the end point of the titration. Potassium ferricyanide reacts with ferrous ions to produce an intense deep colour due to the formation of ferrous ferric cyanide complex



Therefore when a drop of titrated solution is treated with a drop of potassium ferricyanide solution, blue colour is developed as long as ferrous ions are present in the soln. At the end point when ferrous ions are completely converted into ferric ions, a drop of the titrated soln does not develop blue colour with the indicator.

Procedure:Part A: Preparation of standard $K_2Cr_2O_7$ soln

weigh accurately about 1.0g of $K_2Cr_2O_7$ crystals into a 250ml standard flask. Dissolve the salt in little distilled water. Make up the solution to the Mark and shake well for uniform concentration.

Part B: Estimation of iron:

Pipette out 25.0 ml of haematite soln into a clean conical flask. add 5ml of 4N HCl and Heat to boiling. To the hot solution, add $SnCl_2$ solution drop wise from a burette till the yellow color is discharged completely. Add two drops in excess. Cool the soln and add 5 ml $HgCl_2$ solution at a stretch and shake well. A silky white precipitate should be obtained. Add two test tubes of distilled water.

Place a no. of drops of freshly prepared potassium ferricyanide soln on a wax paper. Add 1ml of potassium dichromate soln from the burette into the conical flask and shake well. with the help of a thin glass rod take a drop of the titrated solution and bring it in contact with an indicator drop turns blue. Likewise after each addition of 1ml $K_2Cr_2O_7$ soln, shake the soln well and test the soln drops as before. This titration gives the range of the end point.

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EXP. NO. 03

Again pipette out 25.0 ml of the haematite solution into a conical flask. Add 5 ml of $4N HCl$ and add $SnCl_2$ and $HgCl_2$ as before. Add 2 test tubes of distilled water. Now add most of the potassium dichromate soln req at a stretch and further titrate drop wise testing as explained earlier, till no blue colour is developed with the indicator. Repeat for agreeing values.

Result: Percentage of Iron in given Haematite ore is = 15.72 %.

Record	:	10
observation	:	10
vi va	:	10
Total	:	30

~~10/09/21~~ 09/09/21

$W_1 = 7.838 \text{ g}$

$W_2 = 3.508 \text{ g}$

Weight of EDTA = 4.33 g

Part A: $M_{\text{EDTA}} = \frac{(W_1 - W_2) \times 4}{\text{Weight of EDTA} (372.24)}$

$$= \frac{4.33 \times 4}{372.24}$$
$$= 0.0465 \text{ M}$$

Part B:

Burette: Standard EDTA solution
conical flask: 25.0 ml of hard water + 2 ml $\text{NH}_4\text{OH} - \text{NH}_4\text{Cl}$ buffer

Indicator: A pinch of Eriochrome Black T
Colour change: wine red to blue.

Burette reading in ml	I	II	III
Final burette reading	5.2	5.2	5.2
Initial burette reading	0	0	0
Volume of EDTA in cm^3	5.2	5.2	5.2

Concordant value of EDTA = 5.2 ml.

DATE 1/09/21

EXPT. TITLE:

EXP. NO. 04

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Estimation of Hardness of water:

Aim: To estimate the total hardness of the given water sample.

Principle: Hardness of water is due to the presence of dissolved salts of calcium, magnesium and other metal ions. Hard water does not give lather easily with soap. As the ions causing hardness react with soap to form insoluble soap. Temporary hardness is due to bicarbonates of calcium and magnesium, which can be removed by boiling. On boiling, the bicarbonates are converted into insoluble carbonates of calcium and magnesium, which can be removed by chemical treatment. The sum of temporary hardness and permanent hardness is known as total hardness. The hardness is expressed in ppm equivalents of calcium carbonate (CaCO_3) mg/L .

Hardness is determined by titrating a known volume of water sample with EDTA, using Eriochrome Black T (EBT) as indicator, in presence of ammonium hydroxide ammonium chloride buffer (pH 10). Ethylene Diamine Tetra acetic acid (EDTA) is slightly soluble in water. Hence its disodium salt, which is very much soluble in water, is generally used. EDTA reacts quantitatively with metal ions to form a soluble and stable complex.

When a few drops of the indicator EBT are added, it forms a wine red coloured complex with the metal ions.

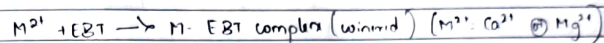
$$M_{\text{CaCO}_3 \text{ in hard water}} = \frac{(MV)_{\text{EDTA}}}{V_{\text{Hard water}}}$$

$$= \frac{0.0465 \times 5.2}{25}$$

$$= 9.672 \times 10^{-3}$$

$$\begin{aligned} \text{Weight of CaCO}_3 / \text{litre of hard water} &= M_{\text{CaCO}_3} \times \text{Molecular wt of CaCO}_3 \\ &= 9.672 \times 100 \times 10^{-3} \\ &= 0.9672 \text{ g (say)} \end{aligned}$$

$$\begin{aligned} \text{wt of CaCO}_3 / \text{million cm}^3 &= \frac{0 \times 10^6}{1000} \\ &= \frac{0.9672 \times 10^6}{1000} \\ &= 967.2 \text{ ppm.} \end{aligned}$$



During titration, EDTA reacts preferentially with free metal ions present in the soln. Near the end point, when the free metal ions are exhausted in soln, further addition of EDTA dissociates the M-EBT complex, consumes the metal ions and releases free indicator. Thus the indicator EBT is suitably selected as it shows a colour change from wine red to blue blw pH 9-11.

Procedure:

Part A: Preparation of standard soln of EDTA:

weigh accurately the given amount of EDTA and transfer into a 250ml standard flask. Dissolve completely with 5ml of NH_3 and sufficient amount of water. Make up the soln to the mark and shake well for concentration.

Part B: Estimation of hardness of water:-

Pipette out 25.0 ml of hard water into a clean conical flask. Add 2ml of buffer soln and a pinch of Eriochrome Black T indicator. Titrate against EDTA soln taken in the burette until the colour

of the soln c

of the soln changes from wine red to blue.
Repeat the titration for concordant values.

Result: Total hardness in the given sample of water

= 967.2 ppm of CaCO_3

Record	:	10
observation	:	10
viva	:	10
Total	:	30

09/09/21

Part A:

Weight of the bottle + EDTA = $W_1 = 7.838 \text{ g}$

Weight of the empty bottle = $W_2 = 3.508 \text{ g}$

Weight of EDTA = $(W_1 - W_2) = 4.33 \text{ g}$

$$M_{\text{EDTA}} = \frac{\text{Wt of EDTA} \times 4}{\text{Mol. wt of EDTA (372.24)}}$$

$$= \frac{4.33 \times 4}{372.24}$$

$$= 0.0465 \text{ M}$$

Part B:

Burette: standard EDTA

conical flask: 25.0 ml of cement solution + 10 ml of glycerol and diethyl amine mixture + 10 ml of NaOH shake well for 3-5 min + add of distilled water

Indicator: A pinch of Patton and Ruders indicator
colour change: Wine red to Blue.

Burette reading in ml	I	II	III
Final Burette reading	5.3	5.4	5.4
Initial Burette reading	0	0	0
Volume of EDTA (ml)	5.3	5.4	5.4

Concordant Value = $V_1 = 5.4 \text{ ml}$

DATE: 1/09/21
EXP. NO.: 05

EXPT. TITLE:

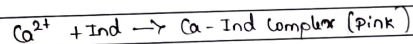
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Estimation of Calcium oxide in cement

Aim: To estimate calcium oxide in the given sample of cement
Soln

Principle: Cement mainly contains silica and also compounds of calcium, aluminium, magnesium and iron. When cement dissolved in acid, silica remains undissolved. On treating with ammonia, aluminium and iron can be precipitated as their hydroxides and separated. The provided solution of cement, therefore, contains calcium and magnesium ions.

To estimate the calcium content in the given solution, a known volume of it is titrated with standard EDTA solution. The interference of magnesium ions is avoided by participating them as hydroxides. This is done by adding 4N NaOH and pH of the soln is maintained 12.5 by adding 5 ml of diethyl amine to the cement solution. The iron mixture containing calcium can be titrated with EDTA by using Patton and Ruders indicator. The indicator combines with calcium ions forming a pink coloured complex.



Near the end point, when free calcium ions are exhausted in the solution, further addition of EDTA dissolves

Wt of the cement given in 250ml soln = $W = 1.28g$

$$(MV)_{EDTA} = (MV)_{CaO \text{ in cement solution}}$$

$$M_{CaO} \text{ in cement} = \frac{MV_{EDTA}}{V_{\text{cement soln}}} = \frac{0.0465 \times 5.4}{25} = 0.01M$$

$$\begin{aligned} \text{Wt of CaO/litre of cement solution} &= M_{CaO} \times \text{Mol Wt of CaO} \\ &= 0.01 \times 56 \\ &= 0.56g \text{ (say a)} \end{aligned}$$

$$\begin{aligned} \text{Wt of CaO/250ml of cement} &= \frac{a}{4} = \frac{0.56}{4} \\ &= 0.14g \text{ (say b)} \end{aligned}$$

$$\begin{aligned} \text{Percentage of CaO in cement sample} &= \frac{b \times 100}{W} \\ &= \frac{0.14 \times 100}{1.28} \\ &= 10.9375\% \end{aligned}$$

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Co-Indicator complex. Consumes the calcium ions and releases the indicator, which is blue in colour. Therefore, the colour change is pink to blue.

A sharper end point made obtained by adding 5ml of 2:1 glycerol reduces adsorption of the complex on the surface of the precipitate.

Procedure:

Part A: Preparation of standard solution of EDTA:

weigh accurately about 4.5g of EDTA and transfer into a 250ml standard flask. Dissolve it completely with 5ml of NH_3 and sufficient amount of water. Make up the solution to the mark and shake well for uniform concentration.

Part B: Estimation of calcium oxide in cement solution.

Pipette out 25.0ml of the cement soln into a clean conical flask. To the flask add 10ml of glycerol and diethyl mixture. Then add 10ml of 4N NaOH soln. Mix the soln thoroughly for 3-5 min. Titrate against the EDTA solution taken in the burette till the colour changes from wine red to blue. Repeat for agreeing values.

Result: Percentage of CaO in cement sample = 10.9375%

100
09/09/21

Record	10
Observation	10
Viva	10
Total	30

KSIT

Observation and calculation:

Part A: Preparation of bleaching powder solution
Preparation of standard $\text{Na}_2\text{S}_2\text{O}_3$ solution

Wt of weighing bottle and $\text{Na}_2\text{S}_2\text{O}_3$ salt: 22.002g

Wt of empty weighing bottle: 19.743g

Wt of $\text{Na}_2\text{S}_2\text{O}_3$ salt: 2.259g

N $\text{Na}_2\text{S}_2\text{O}_3$: $\frac{\text{Wt of } \text{Na}_2\text{S}_2\text{O}_3 \text{ salt}}{\text{Eq. wt of } \text{Na}_2\text{S}_2\text{O}_3 (248.2)} \times 4$

$$= \frac{2.259 \times 4}{248.2} = \frac{9.036}{248.2}$$

$$= 0.0364 \text{ N} = x$$

Burette reading in ml	I	II	III
Final burette reading	0.3	0.3	0.4
Initial burette reading	0	0	0
Vol of $\text{Na}_2\text{S}_2\text{O}_3$ consumed (ml)	0.3	0.3	0.4

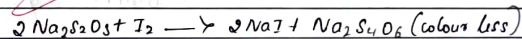
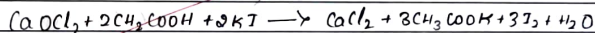
Concordant volume $V = 0.4 \text{ ml}$.

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Determination of chlorine in a given bleaching powder by Iodometric method.

Principle:

Available chlorine sample of bleaching powder is estimated volumetrically. A known mass of bleaching powder as a suspension of water is treated with excess of water is treated with excess of potassium iodide solution. The soln is then acidified with a strong soln of acetic acid. The liberated iodine is titrated against standard sodium sulphate soln using starch as indicator.



Procedure:

part A: Preparation of sodium Thiosulphate solution:

weigh exactly the given amount of $\text{Na}_2\text{S}_2\text{O}_3$ crystals and transfer on to the funnel placed on a clean 500ml volumetric flask. Dissolve it in small quantities of deionized water and make it up to the mark and mix well to get uniform concentration.

Part B: Determination of % of chlorine in the given bleaching powder sol:

Burette: standard sodium thiosulphate solution

Conical flask: 25 cm³ bleaching powder solution + 1 l.t. dist water + 1/4 l.t. acetic acid + 5 ml 10% KI.

Indicator: 2-3 drops of freshly prepared starch

End point: Disappearance of dark blue colour.

1000 cm³ of 1N $\text{Na}_2\text{S}_2\text{O}_3$ solution = 35.5g of chlorine

$$\begin{aligned}\text{Therefore } V \text{ cm}^3 \text{ of } 1N \text{ } \text{Na}_2\text{S}_2\text{O}_3 &= \frac{X \times V \times 35.5}{1000} \\ &= \frac{0.03 \times 0.4 \times 35.5}{1000} \\ &= 0.0004 \text{ (a.g.)}\end{aligned}$$

Wt of bleaching powder taken = $W = 0.035 \text{ g}$

$$\begin{aligned}\text{Percentage of Chlorine} &= \frac{a \times 100}{W} \\ &= \frac{0.0004 \times 100}{0.035} \\ &= 1.1428 \%\end{aligned}$$

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Part B:

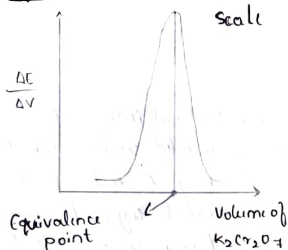
25 cm³ of the prepared soln is pipetted out into a clean conical flask followed by one test tube of distilled water. Add 1/4 test tube of glacial acetic acid followed by 5 ml of 10% potassium iodide. The liberated iodine is titrated against standard sodium thiosulphate solution which is taken in burette till a pale yellow colour is obtained, 2-3 drops of freshly prepared starch indicator is added. The resulting blue soln obtained is titrated against the same soln till the blue colour just disappears. The titration is repeated for concordant values.

Result: The percentage of the available chlorine in the given sample of Bleaching powder is = 1.1428 %

Record :	10
Viva :	10
Observation :	10
Total :	30

per cent of sol.

Graph:

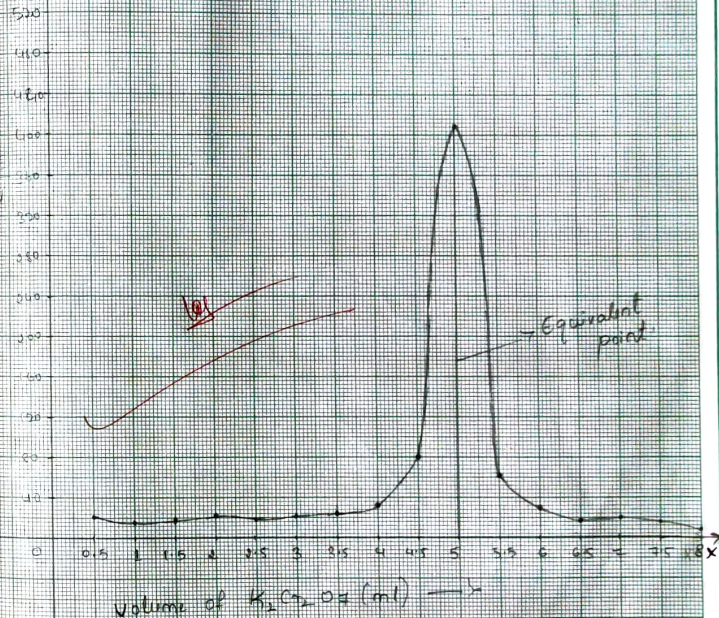


volume of $\text{K}_2\text{Cr}_2\text{O}_7$ solution
 pipetted out = 25 ml
 Normality of $\text{K}_2\text{Cr}_2\text{O}_7 = 0.05\text{N}$

Volume of $\text{K}_2\text{Cr}_2\text{O}_7$ (V) ml	Emf E (mv)	ΔV ($V_2 - V_1$) ml	ΔE ($E_2 - E_1$)	$\Delta E / \Delta V$ (mv/ml)
0.0	165	---	---	---
0.5	176	0.5	11	22
1.0	184	0.5	8	16
1.5	193	0.5	9	18
2.0	203	0.5	10	20
2.5	212	0.5	9	16
3.0	222	0.5	10	20
3.5	234	0.5	12	24
4.0	252	0.5	18	36
4.5	292	0.5	40	80
5.0	516	0.5	224	448
5.5	547	0.5	31	62
6.0	562	0.5	15	30
6.5	571	0.5	9	18
7.0	581	0.5	10	20
7.5	589	0.5	8	16
8.0	593	0.5	4	8

Scale:

X-axis: 1 cm = 0.5 ml
 Y-axis: 1 cm = 40 mv/ml



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EXP. NO. 07

Potentiometric Titration of FAS vs $K_2Cr_2O_7$

Aim: To determine the strength of FAS solution potentiometrically, by using platinum calomel electrode combination in a manner similar to acid-base neutralizations. For the rxn.

Reduced form $\rightarrow$ oxidised form + nelectrons

The potential is given by Nernst equation

$$E = E^{\circ} + \frac{0.0591}{n} \log \frac{[\text{oxidised form}]}{[\text{Reduced form}]}$$

when E° is the standard potential of the system. The potential of the system thus controlled by the ratio of concentration of the oxidized to that of the reduced species present. As the reactions proceeds the ratio increases and hence the potential also increases more rapidly in the vicinity of the end point of titration. A lot of change in potential against volume is characterized by a sudden change of potential at the equivalent point. At the end point potential is determined by

(since $[Fe^{2+}] = 0$)

$$E = 0.7 : E = 1.0V, E = 1.7V$$

Hence there is large increase in potential at the end point.

$$N_{FAS} = \frac{(NV)_{K_2Cr_2O_7}}{V_{FAS}}$$

$$= \frac{0.05 \times 5}{0.5}$$

$$= 0.01 N$$

Amount of FAS per litre = $N_{FAS} \times \text{Eq. wt of FAS (39.2)}$

$$= 0.01 \times 39.2$$

$$= 3.92 \text{ g (say a)}$$

39.2 g of FAS contain 55.85 g Fe

$$x \text{ g of FAS contains } = \frac{55.85 \times x}{39.2}$$

$$= \frac{55.85 \times 3.92}{39.2}$$

$$= 0.5585 \text{ g.}$$

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Pipette out 25.0 cm³ of ferrous ammonium sulphate soln into a beaker. Add 2 test tube of dilute sulphuric acid. Immerse the platinum-calomel electrode assembly into the beaker containing the solution of $K_2Cr_2O_7$. Now add 0.5 ml of $K_2Cr_2O_7$ from the volumetric bottle. Allow the soln to mix well and measure the potential. Take about 5-6 more readings.

Determine the end point by plotting $\frac{\Delta E}{\Delta V}$ against the volume as shown in figure.

Result : Normality of given FAS soln = 0.01 N

$$\text{weight of FAS/ltr} = 3.92 \text{ g/ltr}$$

$$\text{weight of Fe present in one liter} = 0.5585 \text{ g/ltr.}$$

Record	10
observation	10
vi vo	10
Total	30

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Vol of NaOH

conductivity (ohm^{-1}/m)

0	18.4
0.5	17.4
1	16.4
1.5	15.4
2	14.4
2.5	12.4
3	11
3.5	10.0
4	8.5
4.5	7.8
5	6.0
5.5	5.2
6	5.3
6.5	5.4
7	5.6
7.5	5.8
8	6.0
8.5	6.2
9	6.4
9.5	7.3
10	8.1
10.5	8.7
11	9.2
11.5	10
12	10.6
12.5	11.3
13	12
13.5	13.4
14	13

DATE: 6/09/21

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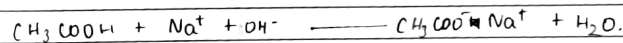
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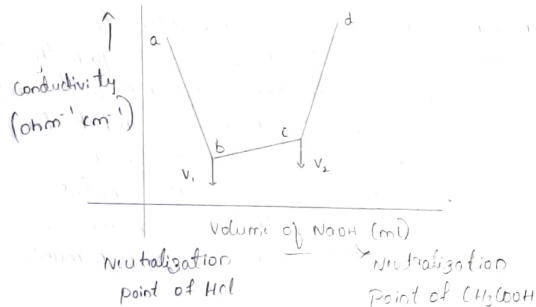
Conductometric Titration

Aim: To determine the strength of mixture of acetic acid and hydrochloric acid using standard NaOH solution conductometrically.

Principle: The determination of end point of a titration by means of conductivity means is called conductometric titration. Conductivity of a solution at a constant temp depends upon the no. of ions per unit volume and change on the ion and their mobility. It is the particular exp. the standard NaOH is added from the burette to measured volume of mixture of acetic acid and hydrochloric acid soln taken in the beaker. in which the conductivity cell is dipped. Once all strong acid is neutralized owing to the conductance increases as the weak acids converted to its ionized salt. Once the neutralization is complete the conductance increases sharply due to high mobility of OH^- ions.

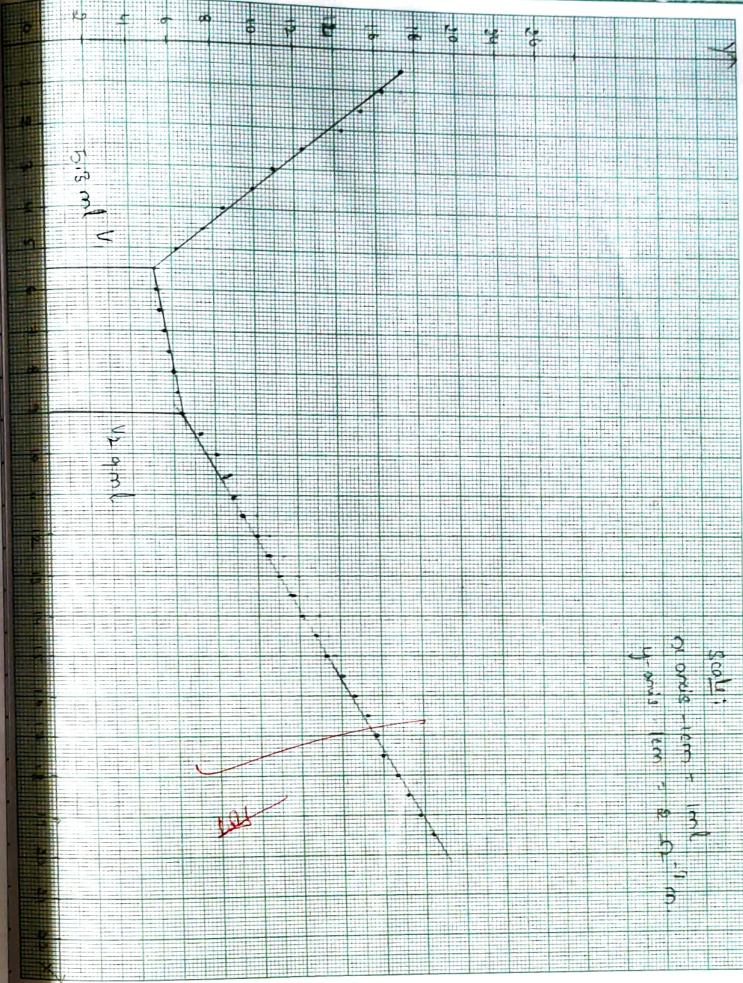


14.5	13.5
15	13.7
15.5	14
16	14.1
16.5	15.3
17	15.7
17.5	16
18	16.7
18.5	17.2
19	17.8
19.5	18.4



observations and calculations:

Strength of Standard NaOH solution	$N_{\text{NaOH}} = 0.5N$
Volume of acid mixture pipetted out	$= 50 \text{ cm}^3$
Volume of NaOH required to neutralize HCl (V_1)	$= 5.3 \text{ cm}^3$
Volume of NaOH required to neutralize both HCl and CH_3COOH (V_2)	$= 9 \text{ cm}^3$
Volume of NaOH required to neutralize CH_3COOH ($V_2 - V_1$)	$= 3.7 \text{ cm}^3$



Procedure :

The conductivity meter is calibrated. 50ml of given mixture of acids is roundown from the burette into clean 100ml beaker and stirred with a magnetic stirrer. The conductivity cell is dipped in the solution. The conductance of soln before addition of base is noted down

The burette is rinsed and filled with std NaOH soln. Small portion of base (2ml) is added each time and the conductance of the solution corresponding to various increments are noted down. A graph is plotted b/w the conductance value (y-axis) and volume of NaOH (x-axis). Two equivalence points are obtained from the graph.

From the volume of alkali consumed the strength of acid can be determined.

Result :

① weight of HCl present in 1dm³ of acid mixture :

② weight of CH₃COOH present in 1dm³ of acid mixture :

~~100~~
0.9

Therefore, the strength of HCl = $N_{HCl} = \frac{N_{NaOH} \times V_1}{50}$

$$= \frac{0.5 \times 5.3}{50}$$

$$= 0.053 N$$

Wt of HCl present in 1 dm^3 of acid mixture = $N_{HCl} \times \text{Eq. wt of HCl}$
 $= 0.053 \times 36.5 \text{ g/dm}^3$
 $= 1.9345 \text{ g/dm}^3$

The strength of CH_3COOH

$$N_{\text{CH}_3\text{COOH}} = \frac{N_{\text{NaOH}} \times (V_2 - V_1)}{50}$$

$$= \frac{0.5 \times 3.7}{50}$$

$$= 0.037 N$$

W of CH_3COOH present in 1 dm^3 of acid mixture :

$$= N_{\text{CH}_3\text{COOH}} \times \text{Eq. wt of } \text{CH}_3\text{COOH}$$

$$= 0.037 \times 60 \text{ g/dm}^3$$

$$= 2.22 \text{ g/dm}^3$$

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Result :

(1) Wt of HCl present in 1 dm^3 of acid mixture =

$$1.9345 \text{ g/dm}^3$$

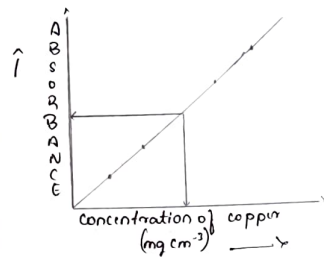
(2) Wt of CH_3COOH present in 1 dm^3 of acid mixture =

$$2.22 \text{ g/dm}^3$$

Record :	10
observation :	10
viva :	10
Total :	30

Net marks 21

Graph:



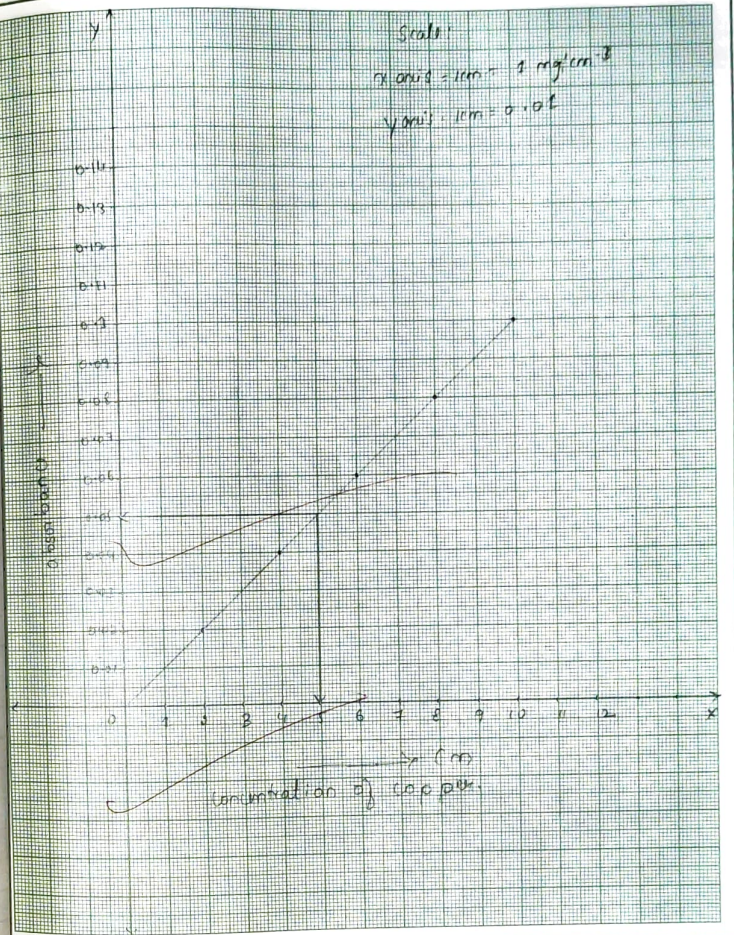
Vol of CuSO_4 in ml	Absorbance
2	0.03
4	0.04
6	0.06
8	0.08
10	0.13
Unknown	0.05

From the graph:

Vol of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ in test solution = 5 ml (say V ml)

Working solution concentration = 4 mg/cc

249.6 mg of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ contains 63.55 mg of Cu^{2+}



Colorimetric Estimation of copper

Aim: To estimate copper colorimetrically using colorimeter.

Principle: When a monochromatic light of intensity I_0 is incident on a transparent medium a part I_a is absorbed, a part I_r is reflected and the remaining part I_t is transmitted

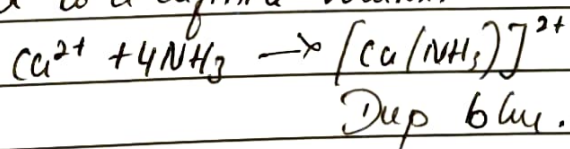
$$I_0 = I_a + I_r + I_t$$

For a glass-air interface I_r is negligible. Therefore,

$$I_0 = I_a + I_t$$

$I_t/I_0 = T$ called the transmittance. $\log 1/T = \log I_0/I_t$ is called the absorbance @ optical density A . The relation b/w absorbance A , concentration C (expressed in mol/ltr) and path length t is given by Beer-Lambert's Law.

A series of standard solns containing cupric ions is treated with ammonia to get deep blue cuprammonium complex and is diluted to a definite volume.



Therefore 4 mg of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ contains $\frac{63.55 \times 4}{249.6}$
 $= 1.02 \text{ mg (say a)}$

1 ml of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ contains 1.02 mg of Cu^{2+}

V ml of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ contains $V \times 1.02 \text{ mg}$ of Cu^{2+}

Since the stock solution is diluted to 50 times

$$\text{Cu}^{2+} \text{ present in test solution} = \frac{V \times a}{50} = \frac{5 \times 1.02}{50}$$
$$= 0.102 \text{ mg}$$

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Procedure:

Part A:

Standardization of the colorimeter:

"Set 310" and "set 100" knobs must be at minimum using an opaque colorimetric tube adjust the "set 310" knob to get 1% transmittance. using blank solution in the colorimetric tube adjust the "set 100" knob to get 100% transmittance without disturbing "set 310" knob. Switch on the colorimetric tube on the O.D. mode.

Part B:

place the given copper sulphate solution in a burette and transfer 2.4, 6.8 and 10 ml of the soln into separate (labelled) 50 cm³ volumetric flasks. Add 2.5 cm³ of ammonia/NH₄OH solution well. To the test soln taken in a 50 cm³ standard flask, add 2.5 ml of ammonia solution and make up to the mark. Mix well. Prepare a blank soln by diluting 2.5 cm³ of ammonia solution in 50 cm³ volumetric flask to the mark with ion exchange water and mix well. After 10 minutes, measure the absorbance of the solutions against blank at 620 nm using a spectrometer.

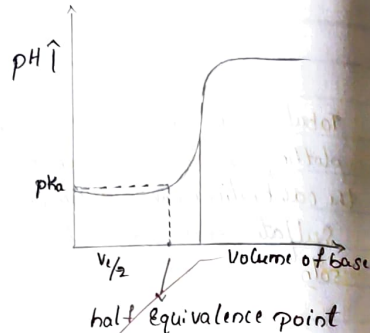
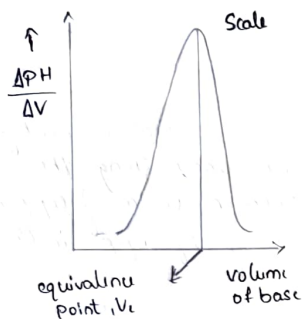
Tabulate the readings as shown. Draw a calibration curve by plotting volume of copper against absorbance. Using the calibration curve determine the unknown volume of copper sulfate and calculate the amount of copper in the soln

Result: Concentration of Cu^{2+} in test solution = 0.109 mg

Record	:	10
observation	:	10
Viva	:	10
Total	:	30

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Observations and Calculations:



Vol of NaOH (V) ml	pH	ΔV (V ₂ -V ₁) ml	ΔpH (pH ₂ -pH ₁)	$\frac{\Delta pH}{\Delta V}$
0.0	2.53	-	-	-
0.5	2.78	0.5	0.25	0.5
1.0	2.97	0.5	0.19	0.38
1.5	3.16	0.5	0.19	0.38
2.0	3.32	0.5	0.16	0.32
2.5	3.49	0.5	0.17	0.34
3.0	3.63	0.5	0.13	0.26
3.5	3.78	0.5	0.16	0.32
4.0	3.99	0.5	0.21	0.42
4.5	4.20	0.5	0.21	0.42
5.0	4.59	0.5	0.39	0.78
5.5	5.22	0.5	0.63	1.26
6.0	11.74	0.5	6.52	12.5
6.5	12.38	0.5	0.64	1.28
7.0	12.65	0.5	0.27	0.54
7.5	12.78	0.5	0.13	0.26
8.0	12.90	0.5	0.12	0.24

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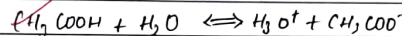
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Determination of Pka value of weak acid

Aim: To determine the pka value of weak acid potentiometrically using pH meter.

Principle: A weak acid like acetic acid dissociates partially in its aqueous soln



The equilibrium constant of dissociation of the acid is known. The acid K_a is given by

$$K_a = \frac{[\text{H}_3\text{O}^+][\text{CH}_3\text{COO}^-]}{[\text{CH}_3\text{COOH}]}$$

The soln is considered to be very dilute and activity of H_2O is assumed to be 1

$$[\text{H}_3\text{O}^+] = K_a \frac{[\text{CH}_3\text{COOH}]}{[\text{CH}_3\text{COO}^-]}$$

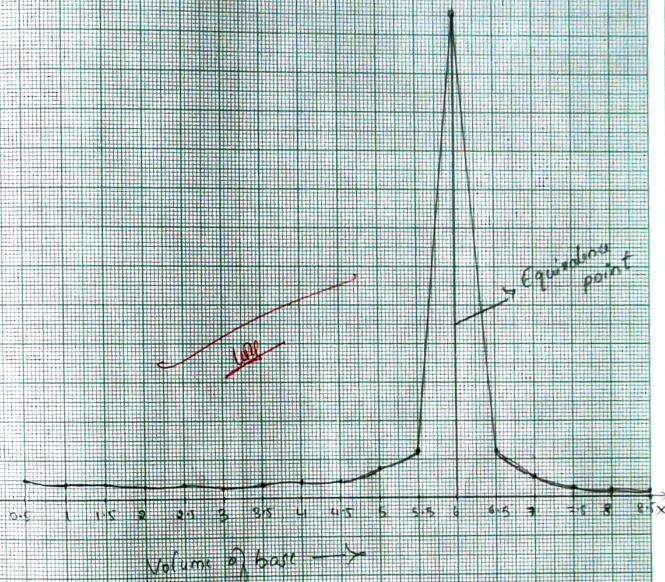
Taking log on both sides

$$-\log[\text{H}_3\text{O}^+] = -\log K_a + \log \frac{[\text{CH}_3\text{COO}^-]}{[\text{CH}_3\text{COOH}]}$$

8.5	12.97	0.5	0.07	0.14
9.0	13.03	0.6	0.06	0.12

$$V_{\text{NaOH}} = 5.6 \text{ ml}$$

$$\frac{V_{\text{NaOH}}}{2} = 2.8 \text{ ml}$$



$$pH = pK_a + \log \frac{[CH_3COO^-]}{[CH_3COOH]}$$

Procedure :

pipette out 25.0 cm³ of given weak acid soln into a beaker. Immerse the glass calomel electrode assembly into the beaker containing the soln. Properly connect to electrodes to the pH meter. Note down the pH of the soln before the addition of NaOH. Now add 0.5 ml of NaOH from the semi burette. Allow the soln to mix well and measure the pH.

Determine the end point by plotting ΔpH against volume of NaOH as shown in the figure. ΔV Now plot the graph taking the volume of NaOH along the x-axis and pH along the y-axis. Then determine the pK_a value from the graph.

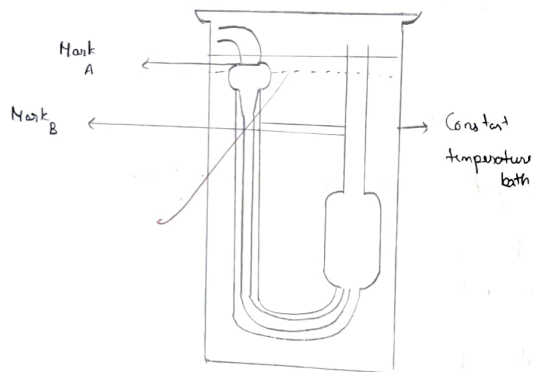
Result: pK_a value of weak acid = 3.55

Record	: 10	
observation	: 10	
viva	: 10	
Total	: 30	09/09/21

Observation and calculations:

Lab temperature: 26°C
 Density of given liquid $d_l = 0.8613 \text{ g/cm}^3$
 Density of water $d_w = 0.9968 \text{ g/cm}^3$
 viscosity of water $\eta_w = 8.737 \text{ millipoise}$

Trial NO	t_1 (secs)	t_2 (secs)
1	34	28
2	33	28
3	33	28
Average	33.33	28



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Coefficient of Viscosity of Liquid.

Aim: To determine the coefficient of viscosity of given liquid using Ostwald's viscometer.

Principle:

V = Volume of liquid

r = radius of tube

l = length of tube

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

η = Co-efficient of viscosity of liquid.

p = pressure difference b/w the 2 ends of tube

If equal volume of 2 different liquids are allowed to flow through the same tube under identical conditions. Then

$$\frac{\eta_l}{\eta_w} = \frac{t_l d_w}{t_w d_l} \quad (1)$$

t_l, t_w - time taken by the test liquid, water chosen to flow through the same distance in the tube respectively.

d_l, d_w - densities of test liquid and water respectively under same room temp respectively.

Coefficient of viscosity of liquid

$$\eta_l = \frac{t_1 d_1}{t_2 d_2} \times \eta_w$$

$$= \frac{33.33 \times 0.8613}{28 \times 0.9968} \times 8.737$$

$$= 8.986 \text{ millipoise.}$$

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η_l, η_w - viscosity co-efficient of test liquid and water resp under same room temp.

Thus co-efficient of viscosity of given test liquid could be found.

Procedure:

Clean the viscometer with water, rinse with acetone and dry it. Fix the viscometer vertically to stand in a constant temp bath using a clamp pipette, transfer 20 cm³ of liquid in to the wider limb. Allow the viscometer to stand for some time so that the liquid attains the temperature of the bath. Suck the water above the upper mark A of the viscometer. Allow it to flow freely through the capillary. When level of water just crosses the lower mark B, note the time of flow in seconds (t_1). Repeat to get agreeing values. Pour out the liquid, rinse the viscometer with acetone and dry it. Repeat the exp with exactly the same volume of water and find the time of flow (t_2 seconds).

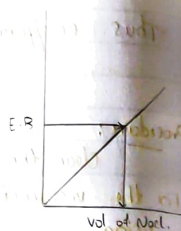
Result: Co-efficient of viscosity of liquid = 8.986 millipoise.

Record :	10
Observation :	10
viva :	10
Total :	30

Observation and calculation

For sodium -

Vol of NaCl	Emission response
2.5	19
5.0	39
7.5	59
10	80
unknown	32



Working solution concentration = 0.1 mg/l

58.5 mg of NaCl contains 23mg of Na

Therefore 0.1 mg of NaCl contains =

$$\frac{23 \times 0.1}{58.5}$$

$$= 0.03931 \text{ (say a)}$$

$$= V \times a$$

$$0.03931 \times 4.2$$

$$= 0.165102 \text{ mg of Na.}$$

DATE 6/09/21

EXPT TITLE

EXP NO 12

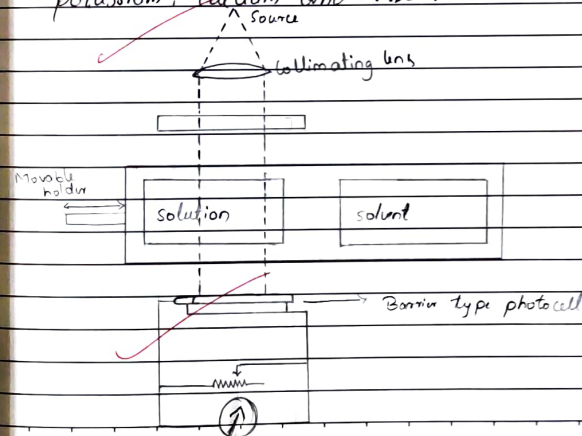
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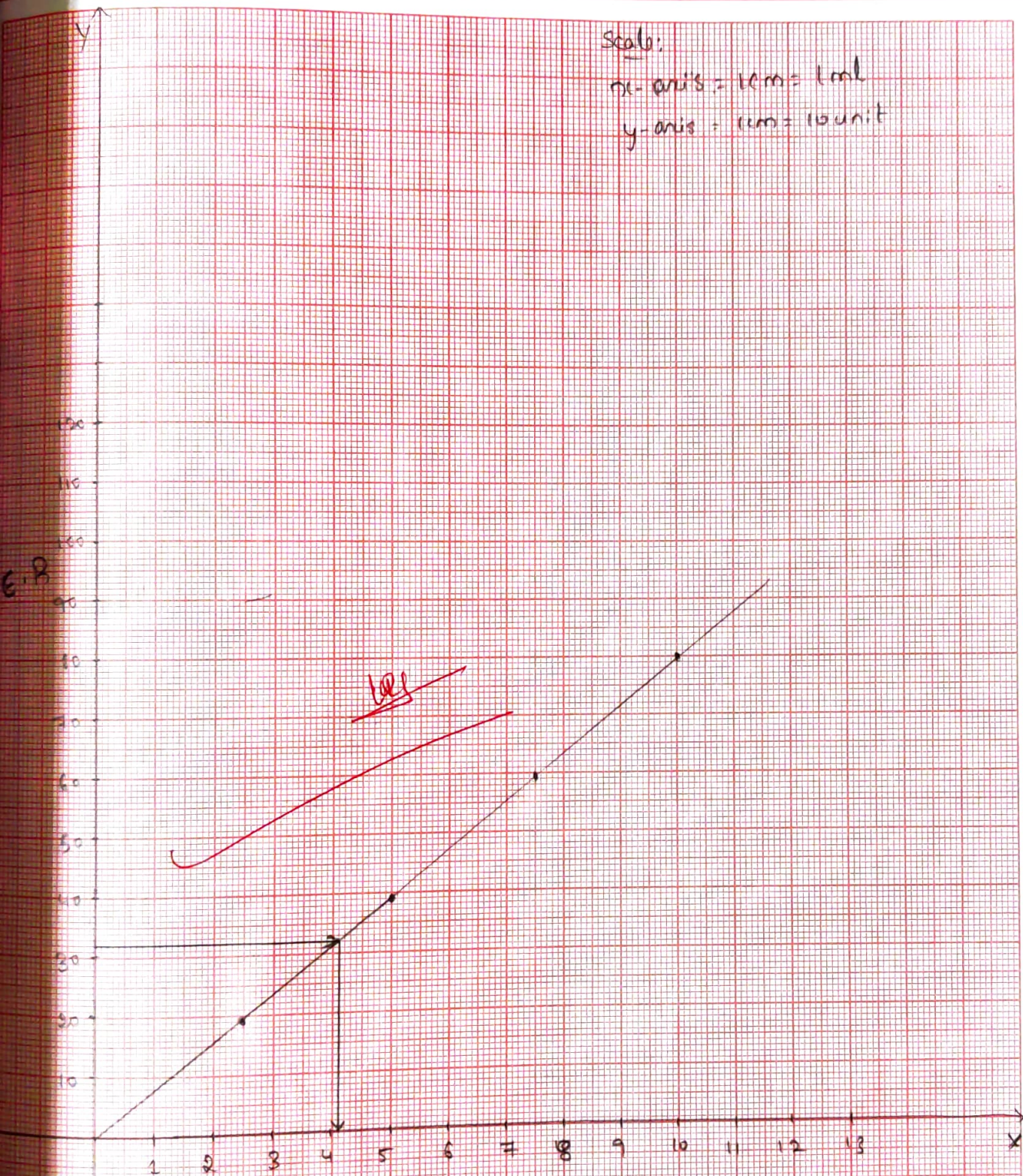
Flame Photometry

Aim: To estimate the sodium and potassium content in the given sample of water by flame photometry.

Principle:

The principle involved in flame photometry is that when a solution containing a metallic compound is aspirated into a flame, a vapour containing metal atoms will be formed. Some of these metal atoms in gaseous state may be raised to an energy level which is sufficiently high to permit the emission of radiation, which is characteristic to the metal under investigation. This method is generally used for the analysis of sodium, potassium, calcium and lithium.





Volume of NaOH $\rightarrow$

Procedure: Pipette out 2.5, 5, 7.5 and 10 ml of standard NaCl soln into four different 50 ml standard flasks. Make the solutions up to the mark and shake well for uniform concentration. Take the first standard soln and aspirate into the flame of the flame photometer. The flame emission intensity is measured by flame photometer using sodium filter (589 nm). The same is repeated for all standard solutions with distilled water into the flame and note down the value of emission response along the y-axis and concentration along the x-axis. Calculate the concentration of the sodium in the water sample. In the same way proceed for potassium.

Result: Amount of Sodium present in the given sample of water = 0.165102

(Amount of Potassium present in the given sample of water = 0.165102)

Record	10
observation	10
viva	10
Total	30

09/09/21