A.P.S COLLEGE OF ENGINEERING

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PARTICULARS OF THE EXPERIMENTS PERFORMED

Name of the Student Aishwarya M Hosur **Course** CSE **Sem** IInd

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REMARKS

15 + 10 = 25

FACULTY

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PARTICULARS OF THE EXPERIMENTS PERFORMED

Name of the Student Aishwarya M Hosur Course CSE Sem 2nd

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REMARKS

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DATE

EXP. NO.

EXPT TITLE

PAGE NO

Observation:

Part A: Preparation of standard solution of disodium salt of EDTA

1. Weight of bottle + EDTA salt = 5.8165 g
2. Weight of empty bottle = 3.7694 g
3. Weight of EDTA salt = 2.0471 g

$$\text{Molarity of EDTA} = \frac{\text{Weight of EDTA salt} \times 4}{\text{Molecular weight of EDTA salt (372.24)}}$$

$$= \frac{2.0471 \times 4}{372.24}$$

$$= 0.0220\ M$$

Part B: Determination of total hardness of water sample

Burette : Standard EDTA solution.

Conical flask: 25 cm^3 water sample + 3 cm^3 of NH_3-NH_4Cl buffer

Indicator: Eriochrome Black T

End point: Wine red to clear blue.

DATE 19/3/24

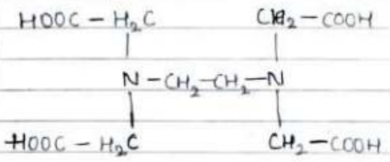
EXP. NO 01

EXPT TITLE ESTIMATION OF TOTAL HARDNESS OF WATER BY EDTA METHOD

PAGE NO 1

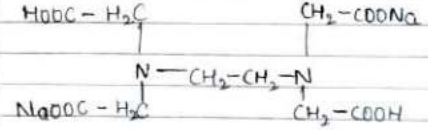
Principle:

Hardness of water is due to the presence of calcium and magnesium salts in water. Ethylenediamine tetraacetic acid (EDTA) forms complexes with a large number of cations including Ca^{2+} and Mg^{2+} ions. Accordingly, it is possible to determine the total hardness of water using EDTA reagent.



$$\begin{array}{ccc} HOOC-H_2C & & CH_2-COOH \\ | & & | \\ & N-CH_2-CH_2-N & \\ | & & | \\ HOOC-H_2C & & CH_2-COOH \end{array}$$

EDTA (H_4Y)



$$\begin{array}{ccc} HOOC-H_2C & & CH_2-COONa \\ | & & | \\ & N-CH_2-CH_2-N & \\ | & & | \\ NaOOC-H_2C & & CH_2-COOH \end{array}$$

Na_2H_2Y

The EDTA molecule (H_4Y) has two easily replacable hydrogen ions and the resulting ion after ionisation may be represented as H_2Y^{2-}. The latter forms complexes with metal ions as follows.

$$M^{2+} + H_2Y^{2-} \longrightarrow MY^{2-} + 2H^+ \quad \ldots\ldots (1)$$

Burette Reading	I	II	III
Final Reading.	14.3	14.4	14.2
Initial Reading	0.0	0.0	0.0
Volume of EDTA run down in cm^3	14.3	14.4	14.2

Calculation:

Volume of EDTA used = 14.4 cm^3

1000 cm^3 of 1M EDTA = 100 g of $CaCO_3$

(Molecular mass of $CaCO_3$ = 100)

14.4 cm^3 of 0.022 M EDTA = $\frac{14.4 \times 0.022 \times 100}{1000 \times 1}$

= 0.0317 C g of $CaCO_3$/25 cm^3 of hard water

∴ Total hardness of the given water sample in parts per million (ppm) = $\frac{C \times 10^6}{25}$ ppm of $CaCO_3$

= 1300 ppm of $CaCO_3$.

Where M^{2+} is Ca^{2+} and Mg^{2+} present in water. Reaction (1) can be carried out quantitatively at a pH of 10 using Eriochrome Black T 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 NH_3-NH_4Cl The hardness of water is usually expressed in terms of ppm (parts per million) of $CaCO_3$. Since EDTA (free acid) is sparingly soluble, its disodium salt, Na_2H_2Y is used for preparing the reagent

Procedure:

Part A: Preparation of standard solution of disodium salt of EDTA

Weigh the weighing bottle containing about 2.5 g of disodium salt of EDTA accurately using an electronic balance and note the weight Transfer the crystals carefully onto a funnel placed over a 250 cm^3 volumetric flask and record the weight of the empty weighing bottle. Pour in exchange water through the funnel allowing all the crystals to run down into the flask. Add 5 ml Ammonia solution. Wash the funnel with ion exchange water and remove the funnel. Dissolve the crystals by swirling the flask gently. Dilute the solution up to the mark with ion exchange water, stopper the flask and mix the solution thoroughly by inverting the flask several times so that a homogeneous solution results.

Part B: Determination of total hardness of water sample

pipette out 25 cm^3 of the given water sample into a clean conical flask. Add 3 cm^3 of NH_3-NH_4Cl buffer and a pinch/ few drop of Eriochrome Black T Indicator. Titrate against standard EDTA solution till the colour of the solution changes from wine red to clear blue. Perform the titration slowly near the end point.

Result:

Total hardness of the given water sample in parts per million (ppm) = 1300 ppm of $CaCO_3$

10

19/03/24

Observation:

Part A: Preparation of standard Ferrous Ammonium sulphate (FAS) solution (Mohr's salt)

1. Weight of bottle + FAS crystals = 6.8202 g

2. Weight of empty bottle = 3.8121 g

3. Weight of FAS crystals = 3.0081 g

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

$$= \frac{3.0081 \times 4}{392}$$

$$= \underline{0.0307\ N}.$$

Back Titration - Estimation of COD of waste water sample

Burette : Standard FAS Solution

Conical flask : 25 cm^3 of waste water sample + 10 cm^3 of standard potassium dichromate solution + 2l.t of 6N H_2SO_4 + boiling chips, reflux the mixture for half an hour, cool.

Indicator : 2-3 drops of ferroin

End point : Blue green to reddish brown.

DATE 26/3/24

EXP. NO. 02

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

Principle:

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

Waste water contains organic impurities which include straight chain aliphatic compounds, aromatic hydrocarbons, straight chain alcohols, acids and other oxidisable materials. Straight chain compounds, acetic acids etc., are oxidised 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 oxidised only partially by this procedure. This difficulty is overcome by adding mercuric sulphate to the sample.

A known volume of the waste water is refluxed with excess of potassium dichromate solution in sulphuric acid medium and in the presence of Ag_2SO_2 and $HgSO_4$. Potassium dichromate oxidizes all oxidisable impurities. The unreacted dichromate is estimated by titrating against standard FAS solution (Back titration) using

Burette Reading	Trial-I	Trial-II	Trial-III
Final Reading	20.2	19.5	19.5
Initial Reading	0.0	0.0	0.0
Volume of FAS run down in cm^3	20.2	19.5	19.5

Volume of FAS used for back titration = 19.5 cm^3 ... b

(i.e the volume of FAS consumed by unreacted $K_2Cr_2O_7$)

Blank Titration (Value to be given by the examiner)

Volume of FAS used for blank titration = 30.0 cm^3 ... c

Calculation:

1000 cm^3 of 1N FAS solution = 1 equivalent of oxygen = 8 g of oxygen (or 8×10^3 mg of O_2)

$$(c-b)\ cm^3 \text{ of } 10.5 \text{ N FAS solution} = \frac{8 \times 10^3 \times (c-b) \times N \text{ of FAS}}{1000 \times 1}$$

$$= \frac{8 \times 10^3 \times 10.5 \times 0.0307}{1000}$$

= 2.5788 mg of oxygen/ 25 cm^3 of waste water.

ferroin indicator. The amount of $K_2Cr_2O_7$ consumed corresponds to the COD of waste water sample. To calculate this, a blank titration without waste water sample is carried out

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

$$\underset{\text{Bluish green}}{Fe(C_{12}H_8N_2)_3^{3+}} + e \longrightarrow \underset{\text{Reddish brown}}{Fe(C_{12}H_8N_2)_3^{2+}}$$

(1,10-phenanthroline-iron II complex) Ferroin

Procedure:

Preparation of standard Mohr's salt solution (FAS soln)

Weigh about 2.5 grams of Mohr's salt accurately into a 250 cm^3 volumetric flask. Add two test tubes of dilute sulphuric acid and dissolve the crystals. Dilute the solution with ion exchange water up to the mark and shake well.

Back Titration:

Pipette out 25 cm^3 of the waste water sample into a conical flask. Add 10 cm^3 of standard potassium dichromate solution followed by 30 cm^3 of 6N sulphuric acid (containing silver sulphate) with constant shaking. Add boiling chips to the flask. Attach a reflux condenser and reflux the mixture for half an hour. Cool, wash the condenser with ion exchange water and transfer the washings to the flask

∴ 1000 cm^3 of waste water sample contains =

$$= \frac{2.5788 \times 1000}{25}$$

$= \underline{103.152}$ mg of oxygen /dm^3

Add 2-3 drops of ferroin indicator and titrate against standard Mohr's salt solution until the solution turns from blue green to reddish brown Repeat for agreeing values.

Blank Titration:

Pipette out 10 cm^3 of standard pottassium dichromate solution Add 1 test tube of 6N sulphuric acid (containing silver sulphate), reflux for half an hour. Cool and add 2-3 drops of ferroin indicator. Titrate against standard Mohr's salt solution until the colour turns from blue green to reddish brown

Result:

∴ COD of waste water sample = <u>103.152</u> mg of O_2 per dm^3

OBSERVATION:

Part A: Preparation of standard $K_2Cr_2O_7$ solution

1. Weight of bottle + $K_2Cr_2O_7$ crystals = 4.2944 g
2. Weight of empty bottle = 3.7686 g
3. Weight of $K_2Cr_2O_7$ crystals = 0.5258 g

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

$$= \frac{0.5258 \times 4}{49}$$

$$= 0.0429 \text{ N}$$

Part B: Estimation of Iron

Burette: Standard $K_2Cr_2O_7$ solution

Conical flask: 25 cm^3 of TMT bar solution + $\frac{1}{4}$th t.t con HCl, heat + $SnCl_2$ dropwise till colourless + 2 drops in excess, cool + $\frac{1}{4}$th t.t. $HgCl_2$ shake well + 1 t.t. H_2O

Indicator: $[K_3(Fe(CN)_6]$ (external)

End point: No change in the colour of indicator when a drop of reaction mixture is brought in contact with it

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DATE 2/4/2024
EXP. NO 3
EXPT TITLE: ESTIMATION OF PERCENTAGE OF IRON IN TMT BAR USING STANDARD POTASSIUM DICHROMATE SOLUTION

Principle:

Thermo mechanically tested bars (TMT bars) are essential for all type of constuction purposes. It is manufactured using virgin iron ore. TMT bar is electrolysed in a solution of sodium bicarbonate (an inert electrolyte) to form ferric oxide. Ferric oxide so formed is treated with 1:1 HCl to form $FeCl_3$. Since iron in $FeCl_3$ is already in the highest possible oxidation state (Fe^{3+}), it is first reduced to ferrous state by using a slight excess of stannous chloride. The excess of stannous chloride added is destroyed by adding saturated solution of mercuric chloride, since $SnCl_2$ can reduce potassium dichromate. Then, the solution is titrated with standard solution of $K_2Cr_2O_7$ using potassium ferricyanide as an external indicator. The volume of $K_2Cr_2O_7$ consumed is a measure of the amount of iron present in test solution.

$$\underset{\text{Ferric chloride}}{2FeCl_3} + \underset{\text{Stannous chloride}}{SnCl_2} \longrightarrow \underset{\text{Ferrous chloride}}{2FeCl_2} + \underset{\text{Stannic chloride}}{SnCl_4}$$

$$SnCl_2 + \underset{\text{Mercuric chloride}}{2HgCl_2} \longrightarrow \underset{\text{Mercurous chloride}}{Hg_2Cl_2}\downarrow + SnCl_4$$

$$(Fe^{2+} \longrightarrow Fe^{3+} + e^-) \times 6 \longrightarrow (1)$$

$$Cr_2O_7^{2-} + 14H^+ + 6e^- \longrightarrow 2Cr^{3+} + 7H_2O \longrightarrow (2)$$

Burette Reading	Trail-I	Trail-II	Trail-III
Final Reading	15.5	16.3	15.5
Initial Reading.	0.0	0.0	0.0
Volume of $K_2Cr_2O_7$ run down in cm^3	15.5	16.3	15.5

CALCULATION:

Volume of $K_2Cr_2O_7$ used = 15.5 cm^3

Weight of TMT bar in 25 cm^3 = 0.055 g (To be given)

1000 cm^3 of 1N $K_2Cr_2O_7$ = 55.85 g of Fe (1 equivalent of Fe)

$$15.5\ cm^3\ 0.0429\ N\ K_2Cr_2O_7 = \frac{55.85 \times 15.5 \times 0.0429}{1000 \times 1}$$

$$= 0.0371\ g\ \text{of}\ Fe/25\ cm^3\ \text{of solution}$$

∴ Percentage of iron in the given TMT bar solution =

$$= \frac{0.0371\ g \times 100}{\text{Weight of TMT bar}}$$

Adding (1) & (2)

$$6Fe^{2+} + Cr_2O_7^{2-} + 14H^+ \longrightarrow 6Fe^{3+} + 2Cr^{3+} + 7H_2O$$

$$2K_3[Fe(CN)_6] + 3Fe^{2+} \longrightarrow Fe_3[Fe(CN)_6]_2 + 6K^+$$

Ferrous ferricyanide
(Blue)

Procedure:

Part A: Preparation of standard $K_2Cr_2O_7$ solution

Weigh about 1.25 g of potassium dichromate crystals accurately and transfer to a 250 cm^3 volumetric flask. Dissolve in ion exchange water and dilute upto the mark and mix well.

Part B: Estimation of Iron

Pipette out 25 cm^3 of the TMT bar solution into a clean conical flask. Add a quarter test tube of concentrated HCl and heat the solution to boiling. Add stannous chloride to the hot solution drop wise till the yellow solution turns colourless. Add 2 more drops to ensure complete reduction. Cool and add a quarter test tube of mercuric chloride rapidly. A silky white precipitate of mercurous chloride is formed. Add one test tube of iron exchange water. Place a number of drops of freshly prepared potassium ferricyanide Indicator on a wax paper. Add a small quantity of potassium dichromate from a burette to the conical flask containing rust solution and mix well. Remove a drop of the solution from the conical flask and bring

Calculation:

$$= \frac{0.0371 \times 100}{0.055}$$

$$= 6.745 \times 10^1$$

$$= 67.4.$$

it in contact with a drop of the indicator on the wax paper. The colour of the indicator turns blue. Repeat this operation after adding 1 cm³ more of the potassium dichromate solution. The indicator turns blue as long as the titration is incomplete. Continue the titration by adding increments of 1 cm³ of $K_2Cr_2O_7$ at a time and testing as above till a drop of the mixture fails to produce any colour with the indicator drop.
(Note: Clean the glass rod after every test). Repeat the titration by taking another 25 cm³ of the rust solution. This time add most of the potassium dichromate solution required at a stretch and then titrate dropwise. Mix the contents of the flask after every addition and test a drop of the titrated mixtured with a drop of the indicator as described above till the colour of the indicator drop does not change. Repeat for agreeing values.

Result:

Percentage of iron in the given TMT bar solution = 67.4

⑩

02/04/24

OBSERVATION:

	Flow Times
	Time of flow (Seconds)
Water	1. 0 + 55 + 1 = 56 sec
	2. 0 + 57 + 0 = 57 sec.
	3. 0 + 56 + 1 = 57 sec
	Mean time, t_w = 56.6
Given liquid	1. 0+43 + 1 = 44 sec.
	2. 0+45+1 = 46 sec
	3. 0+46 + 1 = 47 sec.
	Mean time, t_1 = 45.6

CALCULATION:

Laboratory temperature = 32 °C

Density of water at 32 °C = 0.9950 g/cm³ ~~centipoise~~

Viscosity coefficient of water at 32 °C = 7.840 ~~g/cm³~~ millipoise

Density of given lubricant at 32 °C = 0.8556 g/cm³

Viscosity coefficient of the given lubricant,

$$\eta_1 = \frac{d_1 \times t_1}{d_w \times t_w} \times \eta_w = 9.43 \text{ millipoise}$$

DATE 19/4/24

EXP NO 4

EXPT TITLE DETERMINATION OF VISCOSITY COEFFICIENT OF A LUBRICANT USING OSTWALD'S VISCOMETER

PAGE NO 10

Principle:

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

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

where 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, η is the coefficient of viscosity of the liquid. If equal volumes of two different liquids are allowed to flow through the same tube under identical conditions, then,

$$\frac{\eta_1}{\eta_w} = \frac{t_1 d_1}{t_w d_w}$$

CALCULATION:

$$\eta_1 = \frac{d_1 \times d_1}{d_w \times t_w} \times \eta_w$$

$$= \frac{0.8556 \times 45.6}{0.9950 \times 56.6} \times 7.840$$

$$= \frac{3.0588 \times 10^2}{5.6317 \times 10^1}$$

$$= 5.4314 \times 10^{-1} \times 10^1$$

= 5.43 millipoise.

The time, t_1 taken by the given liquid to travel through a certain distance in the tube is determined. The time, t_w taken by a standard liquid to travel through the same distance is measured. Knowing the densities, d_1 and d_w of the test liquid and the standard and also the coefficient of viscosity of the standard (η_w). coefficient of viscosity of the test liquid (η_1) can be calculated.

Procedure:

Clean the given viscometer with acetone very well and dry it in a hot air oven and remove the vapours by using suction pump. Fix the viscometer vertically to a stand. Using a pipette, transfer a known volume (say 20 cm^3) of distilled water only into the wider limb. Suck it above the upper mark of the viscometer. Allow it to flow freely through the capillary. When the level of water just cross the upper mark (A) start a stop clock. Stop the stop clock when the water just crosses the lower mark (B). Record the time of flow in seconds (t_w). Repeat for 2 times.

Pour out the water, rinse the viscometer and pipette with acetone and dry as before. Clamp the viscometer vertically to a stand and transfer the same volume (20 cm^3) of the test liquid (lubricant) into the wider limb. As described above, record the time (t_1) taken in seconds by the liquid to flow through the same distance.

Determine the coefficient of viscosity of the lubricant using the relation.

$$\frac{\eta_l}{\eta_w} = \frac{t_l d_l}{t_w d_w}$$

$$\eta_l = \frac{t_l d_l}{t_w d_w} \times \eta_w$$

RESULT: The viscosity coefficient of the given lubricant = 5.43 millipoise

⑩

19/4/24

OBSERVATION:

Volume of copper sulphate solution (cm^3)	concentration of copper = 2.037 mg x Vol. of Solution	Absorbance (optical density)
Blank solution	—	0.00
2.0	4.074	0.09
4.0	8.1480	0.18
6.0	12.22	0.27
8.0	16.296	0.36
10.0	20.370	0.44
Test solution	11.0	0.25

Calculation:

1000 cm^3 of stock solution contains 8 g of $CuSO_4 \cdot 5H_2O$

249.54 g of $CuSO_4 \cdot 5H_2O \equiv 63.54$ g of Cu

8 g of $CuSO_4 \cdot 5H_2O \equiv 63.54 \times 8/249.54 = 2.037$ g of Cu per 1000 cm^3 of stock solution

$\therefore$ 1 cm^3 of $CuSO_4 \cdot 5H_2O \equiv 2.037/1000 = 0.002037$ g of Cu = 2.037 mg of Cu

$\therefore$ Cu present in 'a' cm^3 of test solution = 'a'

5.4 cm^3 × 2.037 mg = 11.0 mg.

DATE 3/5/2024
EXP. NO 5

EXPT TITLE ESTIMATION OF COPPER PRESENT IN ELECTROPLATING EFFLUENT BY OPTICAL SENSOR (COLORIMETRY)

Principle:

When a monochromatic light of intensity I_0 is incident on a transparent medium, a part, I_a of it 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 and therefore,

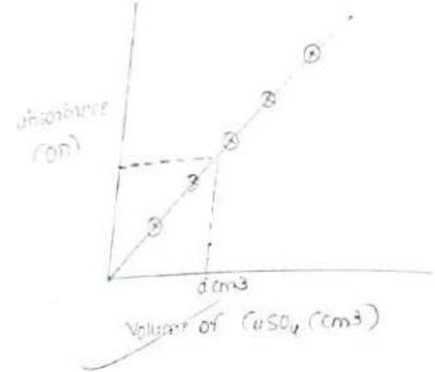
$$I_0 = I_a + I_t$$

$I_t / I_0 = T$ called the transmittance, $\log 1/T = \log I_0/I_t$ is called the absorbance or optical density. The relation between absorbance, A, concentration, c (expressed in mol/dm^3) and path length, t (expressed in cm) is given by Beer - Lambert's law.

$$A = \log I_0/I_t = \epsilon c t$$

where ϵ is the molar extinction coefficient, t is the path length and is a constant for a given substance at a given wavelength. If t, the path length is kept constant, then, $A \propto c$ Hence a plot of absorbance against concentration given a straight line.

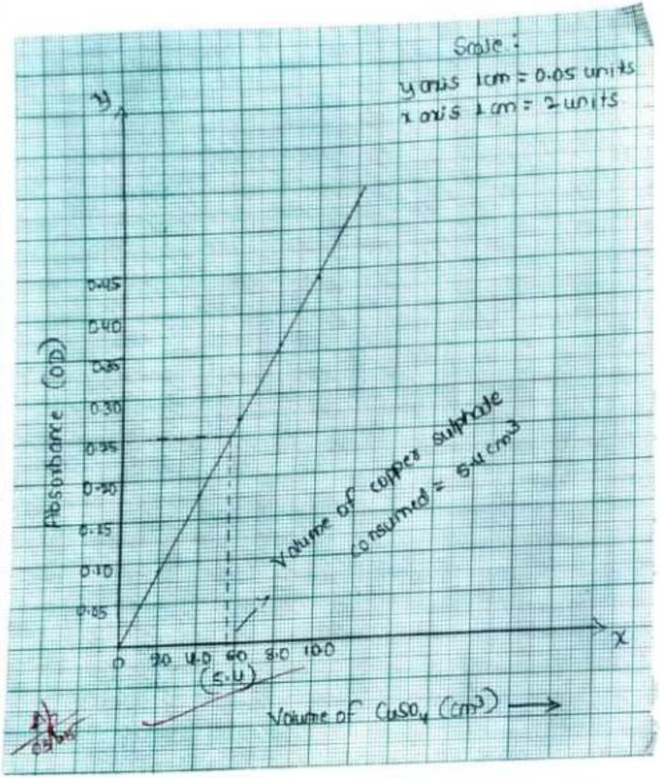
A series of standard solutions of copper sulphate penta hydrate is treated with ammonia to get blue cuprammonium complex, $[Cu(NH_3)_4]^{2+}$ and is diluted to a definite volume. The absorbance of each of these solutions is measured at 620 nm since the complex shows maximum absorbance at this wavelength. The absorbance values are plotted against concentration to get a calibration curve.



The given test solution is treated with strong ammonia and diluted to the same volume as above. The absorbance of this solution at 620 nm is measured and its concentration is determined from the calibration curve.

Procedure:

Transfer the given copper sulphate solution (Stock solution) to a burette and draw out 2, 4, 6, 8 and 10 cm^3 of the solution into 50 cm^3 volumetric flasks. Now collect the test solution from the teacher / examiner, add 5 cm^3 of ammonia solution to each of them. shake well and then dilute up to the mark with ion exchange water. stopper the flasks and mix the solutions well. To the test solution (electroplating effluent) given in a 50 cm^3 measuring flask, also add 5 cm^3 of ammonia solution, shake well and then dilute up to the mark with distilled water and mix well. Prepare a blank solution by diluting only 5 cm^3 of ammonia solution in a 50 cm^3 measuring flask up to the mark with distilled water and mixing well. After 10 minutes, measure the absorbance of the solutions against blank at 620 nm using a photoelectric colorimeter. Tabulate the readings as shown. Draw a calibration curve by plotting absorbance against volume of copper sulphate solution. Using the calibration curve, find ~~th~~ out the volume of copper sulphate solution given i.e. the volume of



test solution and calculate the amount of copper in the given effluent solution.

RESULT:

The weight of copper in the given test solution = 11.0 mg.

10

03/05/24

OBSERVATION:

Volume of NaOH added (cm^3)	pH	ΔV	ΔpH	$\frac{\Delta pH}{\Delta V}$
0.0	3.65	—	—	–
0.5	3.84	0.5	0.19	0.38
1.0	3.98	0.5	0.14	0.28
1.5	4.12	0.5	0.14	0.28
2.0	4.28	0.5	0.16	0.32
2.5	4.48	0.5	0.2	0.4
3.0	4.68	0.5	0.2	0.4
3.5	4.88	0.5	0.2	0.4
4.0	5.16	0.5	0.28	0.56
4.5	5.70	0.5	0.54	1.08
5.0	9.80	0.5	4.1	8.2
5.5	10.98	0.5	1.18	2.36
6.0	11.28	0.5	0.3	0.6
6.5	11.38	0.5	0.1	0.2
7.0	11.52	0.5	0.14	0.28
7.5	11.66	0.5	0.14	0.28
8.0	11.78	0.5	0.12	0.24

DATE 17/5/2024
EXP. NO. 6

EXP. TITLE
DETERMINATION OF pKa VALUE OF VINEGAR USING pH SENSOR

Principle:

The strength of an acid is experimentally measured by determining its equilibrium constant or dissociation constant (Ka) Since strong acids are strong electrolytes. they are ionised almost completely in aqueous solutions. It is not meaningful to study the ionic equilibrium of strong acids and calculate their equilibrium constants as the unionised form is present to such a small extent. Hence, the study of ionic equilibrium and calculation of K is applicable only to weak acids. For eg. Acetic acid (vinegar) ionises feebly as, $CH_3COOH (aq) + H_2O (l) \longrightarrow H_3O^+ (aq) + CH_3COO^- (aq)$

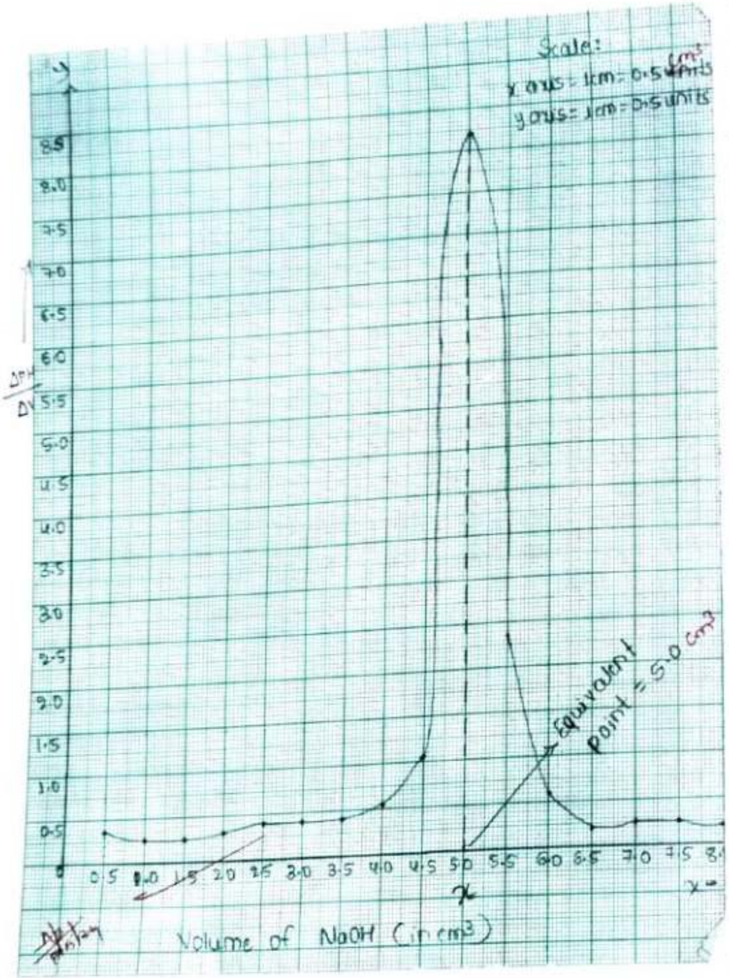
$$K_a = \frac{[H_3O^+][CH_3COO^-]}{[CH_3COOH]}$$

pKa is a modern method of expressing acid strengths.

$$pKa = -\log_{10} Ka$$

pKa is determined by measuring the changes in pH of acid solution at different amounts of the base added

During the titration of an acid with a base, the pH of the solution rises gradually at first, then more rapidly and until at the equivalence point, there is a very sharp increase in pH for a very small quantity of added base Once past the equivalence point, the pH increases only slightly on addition of excess base. The titration curve is obtained by plotting changes in pH at different amounts of the base added and the equivalence point is determined



According to Henderson- Hasselbalch equation.

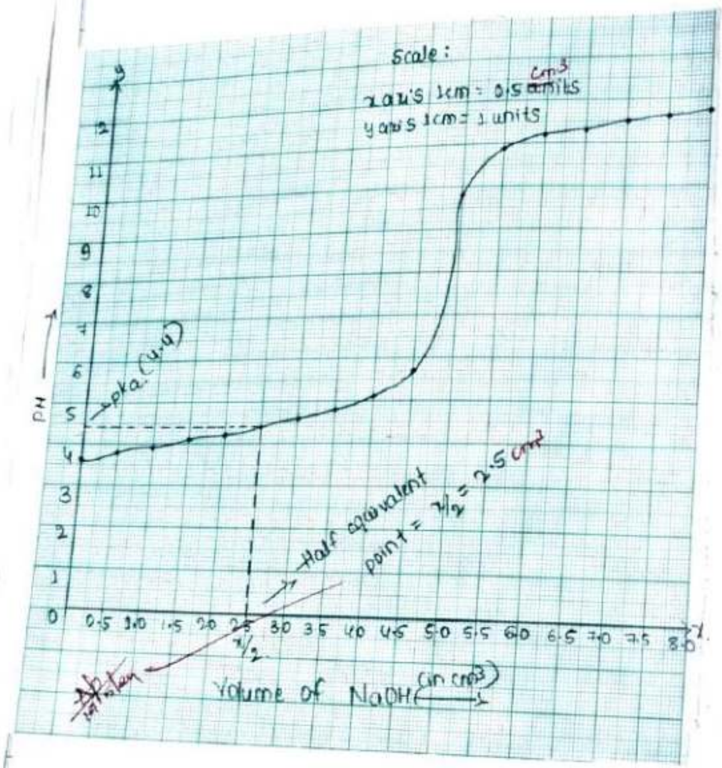
$$pH = pK_a + \log_{10}\frac{[Salt]}{[Acid]}$$

At half equivalence point, [Salt] = [acid] and therefore, pH at half equivalence point gives the pKa of weak acid.

Procedure:

Pipette out 50 cm³ of the given vinegar into a 100 cm³ beaker. Immerse a glass electrode - calomel electrode assembly into the acid and connect the cell to a pH meter. Measure the pH of the acid. Fill a burette with the base (sodium hydroxide). In the beginning, add large increments of (say 0.5 cm³) of the base to the acid. Stir the solution thoroughly and measure the pH after each addition. When the pH begins to show a tendency to increase rapidly, add only small increments (say 0.5 cm³) of the base and measure the pH after each addition.

Plot a graph of pH (ordinate) against the volume of sodium hydroxide added (absissa). Determine the equivalence point and hence the pH at the half equivalence point. This gives the pKa value of the acid.



Result:

1. Equivalence point = x = 5.0 cm³

2. Half equivalence point = x/2 = 2.5 cm³

3. pH at half equivalence point = pKa = 4.4

4 pKa of the given weak acid (vinegar) = pH at half equivalence point = 4.4.

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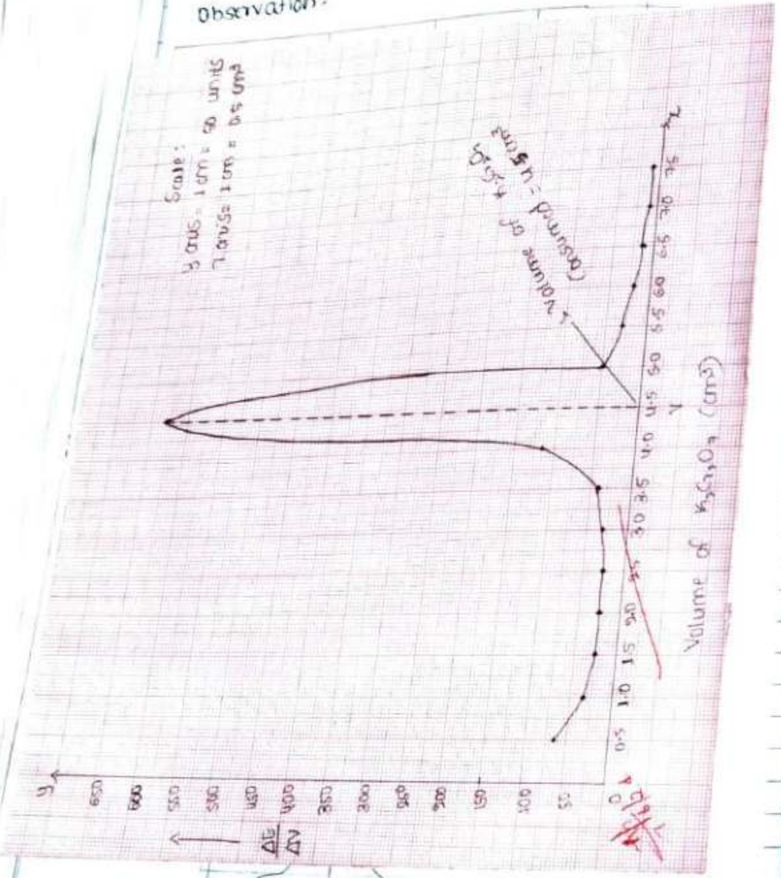
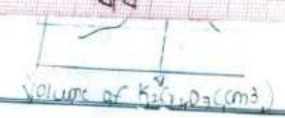
Observation:





Principle:

The procedure of using a measurement of emf to determine the concentration of ionic species in solution is referred to as potentiometry. The principle involved in potentiometric titration is the measurement of emf between two electrodes; an indicator electrode, the potential of which is a function of the concentration of the ion to be determined and a reference electrode (calomel electrode) of constant potential. In these titrations, the measurements of emf are made while the titration is in progress. The equivalence point of the reaction is revealed by a sudden change in potential in the plot of emf readings against the volume of titrant.

The ~~best~~ determining factor in oxidation-reduction reactions is the ratio of the concentration of the oxidised and reduced forms of certain ion species. For the reaction:

$Cr^{6+} + 3e^- \rightarrow Cr^{3+}$, the potential, E acquired by the indicator electrode at 25°C is given by

$$E = E^\circ + \frac{0.0591}{n} \log_{10} \frac{[Cr^{6+}]}{[Cr^{3+}]} \qquad n = 3$$

The potential of the immersed electrode is thus controlled by the ratio of these concentrations. During the oxidation of a reducing agent or the reduction of an oxidising agent the ratio, and therefore the potential, changes more rapidly in the vicinity of the end point.

Calculation:

Normality of $K_2Cr_2O_7$ = 0.5 N (To be given)

Volume of $K_2Cr_2O_7$ added = 4.5 V cm³ (Equivalence point from graph)

Volume of FAS used = 50 cm³

$$\therefore \text{Normality of FAS} = \frac{(N \times V) \text{ of } K_2Cr_2O_7}{\text{Vol. of FAS}}$$

$$= \frac{0.5 \times 4.5}{50}$$

$$= 0.045 N$$

∴ Weight of FAS per litre = Normality × Equivalent Wt. of FAS

$$= 0.045 \times 392$$

$$= \underline{17.64} \text{ g}$$

$$\therefore \text{Weight of FAS}/50 \text{ cm}^3 = \frac{17.64 \times 50}{1000}$$

$$= \underline{0.882} \text{ g}$$

Procedure:

Pipette out 50 cm³ of ferrous ammonium sulphate solution into a 250 cm³ beaker. Add two test tube of dilute sulphuric acid (2.5M). Place the electrode assembly (platinum electrode as indicator electrode and a SCE as reference electrode) in the beaker and connect the same to the pH / Potentiometer. Read the potential difference between the electrodes with the aid of the meter (by using the meter in millivolt mode). and record it. Add 0.5 cm³ of $K_2Cr_2O_7$ solution from a burette, mix the contents and after waiting for a half minute, measure the emf of the cell. Repeat the addition of 0.5 cm³ portions of $K_2Cr_2O_7$, mixing and measuring the emf after each addition until a sudden change in the emf is observed. Henceforth, add the solution in portions of 0.5 cm³ and record the readings after each addition. Take 6 more readings.

Plot emf (ordinate) versus volume of $K_2Cr_2O_7$ added (abscissa). Draw a smooth curve through the points. The equivalence point is the volume corresponding to the steepest portion of the curve. Locate the equivalence point of titration by plotting ΔE/ΔV (ordinate) versus volume of titrant, (abscissa) There is a maximum in the plot at the equivalence point.

RESULT:

Weight of FAS / 50 cm³ = 0.882 g

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Observation:

Vol. of NaOH (cm^3)	Conductance ($ohm^{-1} cm^{-1}$)
0.0	12.8
0.5	11.8
1.0	10.4
1.5	9.2
2.0	8.2
2.5	7.2
3.0	6.0
3.5	4.8
4.0	4.6
4.5	4.8
5.0	5.0
5.5	5.4
6.0	5.6
6.5	5.8
7.0	6.0
7.5	6.2
8.0	6.4
8.5	6.7
9.0	6.9
9.5	7.2
10.0	7.6
10.5	8.4
11.0	9.0
11.5	9.6
12.0	10.3
12.5	10.8
13.0	11.4
13.5	12.0
14.0	12.4
14.5	12.9

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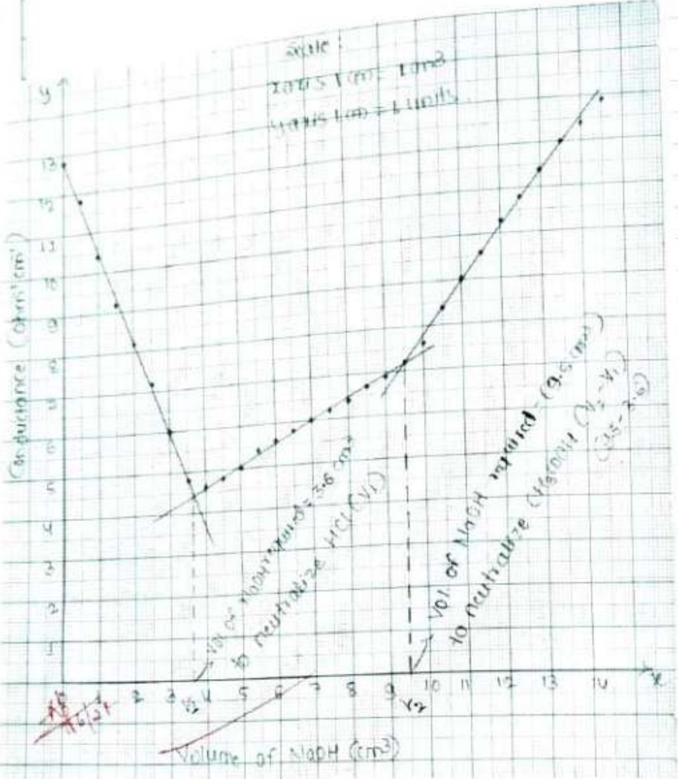
EXPT TITLE CONDUCTOMETRIC ESTIMATION OF AN ACID MIXTUR USING STANDARD NaOH SOLUTION

Theory:

Measurement of conductance can be employed to determine the end point in acid-base titrations. In conductometric titrations, there is a sudden change in conductance of the solution at the equivalence point. The principle underlying conductometric titrations is the substitution of ions of one mobility by ions of another mobility. Therefore, the conductance of a solution depends on the number and mobility of ions. The equivalence point is determined graphically by plotting conductance (ordinate) against titre values (obscissa)

Conductometric titration may be applied for the determination of acids present in a mixture. In the titration of a mixture of a strong acid (HCl) and a weak acid (CH_3COOH) with a strong base (NaOH), the conductance initially decreases upon adding NaOH to acid mixture owing to the substitution of highly mobile H^+ ion (mobility= 350 $ohm^{-1} m^{-1}$) by the less mobile Na^+ ion (mobility: 50 $ohm^{-1} m^{-1}$). This trend continues till all the H^+ ions of HCl replaced i.e., the strong acid is neutralised. Continued addition of NaOH rises the conductance moderately, as the week acid CH_3COOH is converted into its salt, CH_3COONa. Further addition of NaOH rises the conductance steeply due to the presence of free OH^- ions (mobility: 198 $ohm^{-1} cm^{-1}$). The titration curves in the graph given depict the location of the equivalence point.



Estimation of HCl:

Pipette out 50 cm³ of the given acid mixture into a clean 100 cm³ beaker. Dip the conductivity cell in the solution and note down the conductance of the solution i.e, when the volume of NaOH added is zero.

Now add standard NaOH solution from a burette in increments of 0.5 cm³. After each addition, stir the solution gently and note down the conductance. As the titration proceeds, the conductance first gradually decreases and then rises slowly and finally rises sharply. Continue titration until the conductance is more or less the same as it was in the beginning. Plot a graph of conductance on Y-axis versus volume of NaOH on X-axis to get three straight lines as shown in the figure. The point 'V₁' gives the volume of sodium hydroxide needed to neutralize hydrochloric acid and the point 'V₂' gives the volume of NaOH needed neutralize both HCl and CH_3COOH. The difference in the volumes i.e., $(V_2 - V_1)$ gives the volume of NaOH needed to neutralize CH_3COOH.

Estimation of HCl:

Volume of the acid mixture taken = 50 cm^3

Normality of NaOH = 1.0

Volume of NaOH required to neutralise HCl = 3.6 V_1

$$\text{Normality of HCl} = \frac{\text{Normality} \times \text{Volume of NaOH}(V_1)}{50}$$

$$= \frac{1.0 \times 3.6}{50} = \underline{0.072\ N}$$

Therefore, the weight of HCL dm^3 = Normality of HCL × Eq. wt. of

$= 0.072 \times 36.5$

$= \underline{2.628\ g}$

Estimation of CH_3COOH:

Volume of NaOH required to neutralise $CH_3COOH = (V_2 - V_1)\ cm^3$

$9.5 - 3.6 = \underline{5.9\ cm^3}$

$$\text{Normality of } CH_3COOH = \frac{\text{Normality} \times \text{Volume of NaOH}(V_2 - V_1)}{50}$$

$$= \frac{1.0 \times 5.9}{50} = \underline{0.118\ N}$$

Therefore, the weight of CH_3COOH/dm^3 = Normality of CH_3COOH × Eq. of CH_3COOH (60)

$= 0.118 \times 60$

$= \underline{7.08\ g}$

RESULT:

Vol. of NaOH required to neutralise HCl = $\underline{3.6\ cm^3}$

Normality of HCl = $\underline{0.072\ N}$

Weight of HCl = $\underline{2.628\ g}$

Vol. of NaOH required to neutralise $CH_3COOH\ (V_2 - V_1)$ = $\underline{5.9\ cm^3}$

Normality of CH_3COOH = $\underline{0.118\ N}$

Weaight of CH_3COOH = $\underline{7.08\ g}$

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EXPT TITLE:

SYNTHESIS OF IRON OXIDE NANOPARTICLES

In recent years, iron oxide nanoparticles have demonstated great potential in biomedical applications due to their non-toxic role in biological systems. Also, the magnetic and semiconductor properties of iron oxide nanoparticles can lead to multifunctional applications in medicine. These nanoparticles have been developed as antibacterial, antifungal, and anticancer. For cancer treatment and diagnosis, iron oxide nanoparticles have been functionalized with drugs. However, many of those drugs have been related to adverse effects on health. On the other hand, phytochemicals from extracts of plants have been used as an alternative for the functionalization of these nanoparticles preventing negative effects The main advantage of these nanoparticles is the high biodistribution in the organism compared with other drug delivery systems. The magnetism of iron oxide nanoparticles has been used in cancer treatment and diagnosis. for example, thermoablation, hyperthermia and contrast media in magnetic resonance imaging.

Iron oxide nanoparticles are synthesized by precipitation method, which is a simple technique among chemical route This method is given more priority due to its low cost and easy industrialization with improved reproducibility This method offers low temperature growth and scale-up fabrication which yields colloidal solutions with wide range of particle districutions

Principle:

co-precipitation is a very facile and convenient way to synthesize iron oxide nanoparticles (Fe_3O_4) from aquous Fe^{2+}/Fe^{3+} salt solutions by the addition of a base at room temperature or at elevated temperature

Chemicals required:

$FeSO_4 \cdot 2H_2O$ (0.2M), $FeCl_3 \cdot 6H_2O$ (0.1M),
Ammonia solution (3M) and deionized water.

Procedure:

0.2M ferrous sulphate solution and 0.1 M ferric chloride solutions are mixed with 100 ml deionized water in a beaker Ammonia solution (3M) is added dropwise with constant stirring at 1000 rpm using a magnetic stirrer till pH 10. The black precipitate of iron oxide obtained is filtered and dried in hot-air oven at 150-200°C

Reaction:

$$2FeSO_4 + FeCl_3 + 5NH_3 \longrightarrow Fe_3O_4 + (NH_4)_2SO_4 + 3NH_4Cl$$

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EXPT TITLE:
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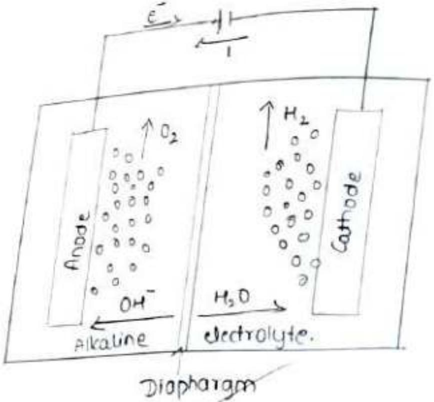
ELECTROLYSIS OF WATER.

Electrolysis of water, also known as electrochemical water splitting, is the process of using electricity to decompose water into oxygen and hydrogen gas by electrolysis Hydrogen gas released in this way can be used as hydrogen fuel, or remixed with the oxygen to create oxyhydrogen gas, which is used in welding and other applications

Electrolysis of water requires a minimum potential differene of 1.23 volts, throught at that voltage external heat is required from thu environment

A DC electrical power source is connected to two plates (typically made from an inert metal such as platinum or graphite) which are placed in the water. Hydrogen will appear at the cathode (where electrons enter the water), and oxygen will appear at the anode. Assuming ideal faradaic efficiency, the amount of hydrogen generated is twice the amount of oxygen, and both are proportional to the total electrical charge conducted by the solution. However, in many cells competing side reactions occur, resulting in different products and less than ideal faradac efficiency.

Electrolysis of pure water requires excess energy in the form of over potential to overcome various activation barriers. Without the excess energy, the electrolysis of pure water occurs very slowly or not at all. This is in part due to the limited self-ionization of water. Pure water has an electrical conductivity about one millionth that of



seawater. Many electrolytic cells may also lack the requisite electrocatalysts. The efficiency of electrolysis is increased through the addition of an electrolyte (such as a salt, an acid or a base) and the use of electrocatalysts.

Alkaline electrolysis of water:

The important components used in the electrolyser are as shown in the figure.

Anode: Nickel metal particles dispersed on porous carbon is used as anode electrocatalyst.

Cathode: Nickel metal particles coated on porous carbon is used as cathode electrocatalyst.

Electrolyte: Aqueous solution of KOH (with 20 to 30 weight % KOH) is used as an electrolyte.

Separator: Porous dense anion exchange membrane is used as the separator. It is a good ionic conductor of hydroxyl ions and bad electronic conductor. It prevents the spontaneous recombination of H_2 and O_2

Deionised water is passed in to the cathode chamber. At cathode water molecules are reduced to hydrogen and hydroxyl ions. The hydroxyl ions move through the separator towards anode and get oxidized to oxygen and water.

Reactions: At cathode: $2H_2O + 2e^- \rightarrow H_2(g) + 2OH^-$

At anode: $2OH^- \rightarrow \frac{1}{2}O_2(g) + H_2O + 2e^-$

Overall reaction: $H_2O \rightarrow H_{2(g)} + \frac{1}{2}O_2(g)$

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