Experiments

List of Experiments

1. To determine the alkalinity of given water sample due to dissolved NaOH & Na_2CO_3 by neutralization titration
2. To determination of temporary and permanent hardness of water sample by complex metric titration using EDTA.
3. Determination of available Iodine in the common salt sample.
4. Determine the percentage of available Chlorine in Bleaching powder sample.
5. To determine the chloride content in given water sample by Mohr's method.
6. Determination of iron (Fe^{2+}) by redox titration using external indicator and internal indicator.
7. Study of variation of viscosity with variation in concentration and determination of intrinsic viscosity.
8. Titration of HCl solution against standard NaOH solution using a pH-meter.
9. To determine the concentration of $KMnO_4$ solution spectrophotometrically.
10. Detection of N, S, Cl, Br and I in organic compounds.
11. Identification of functional group in an organic compound.
12. To prepare phenol formaldehyde resin (Bakelite) and urea formaldehyde resin (UF).
13. Preparation of Alum
14. To determine surface tension of the given liquid at room temperature by Stalagnometer.
15. Separation of red ink and blue ink by paper chromatography.

EXPERIMENT NO 1

Object

(1) Determine acidimetry and alkalimetry of given acid and base

(2) To determine the alkalinity of given water sample due to dissolved NaOH & Na_2CO_3 by neutralization titration.

Theory

Acidimetry is determination of concentration of acid by titrating against a standard solution of base.

Alkalimetry is determination of concentration of base by titrating against a standard solution of acid.

Alkalinity is amount of OH^- in a given water sample which can be increased by hydrolysis or dissociation.

The experiment is based on 'Theory of Indicators'. Phenolphthalein indicator shows a change in color from colorless to pink in pH range 8.0-9.8. This is attained when titration of NaOH against HCl is complete and titration of Na_2CO_3 against HCl is half complete, i.e. Na_2CO_3 is completely converted to $NaHCO_3$.

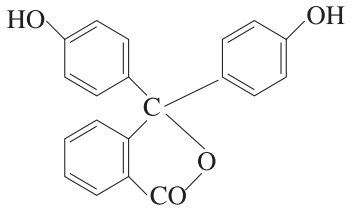
$$NaOH + HCl \rightleftharpoons NaCl + H_2O$$

$$Na_2CO_3 + HCl \rightleftharpoons NaHCO_3 + NaCl$$

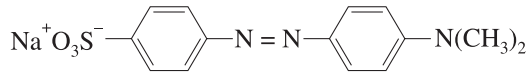
Methyl Orange indicator shows a change in colour from light yellow to red in the pH- range 3.1-4.5. This pH attained when both NaOH and Na_2CO_3 are completely neutralized.

$$NaOH + HCl \rightleftharpoons NaCl + H_2O$$

$$Na_2CO_3 + 2HCl \rightleftharpoons 2NaCl + H_2O + CO_2$$



Structure of Phenolphthalein



Structure of methyl orange

Procedure

Acidimetry Take 10ml of acid in conical flask and base in burette. Indicator used is methyl orange. End point is where pink color is obtained.

Alkalimetry Take 10ml of base in conical flask and acid in burette. Indicator used is phenolphthalein End point where pink color disappears.

Alkalinity Take 10ml of water sample in a 250 ml conical flask with help of a pipette. Add 2-3 drops of phenolphthalein indicator. Titrate this sample against standard HCl solution until the pink

color just disappears. Add 2-3 drops Methyl Orange indicator. Light yellow color appears. Continue the titration with the HCl in burette until the color turns pink.

Note three concurrent values of titre volumes with each indicator. (Never use mean values.)

Observation

Alkalimetry

S.No.	Vol. of acid	Vol. of base

Acidimetry

S.No.	Vol. of acid	Vol. of base

Alkalinity

S. No	Volume of Water Sample (in Pipette) (ml.)	Titre volume of HCl (in burette)	
		With Phenolphthalein (ml.)	With Methyl Orange (ml.)
1.			
2.			
3.			

Result Alkalinity due to NaOH = ppm.

Alkalinity due to Na_2CO_3 = ppm.

Exercise

1. Write the principle of titration.
2. Learn the theory of Indicator. (Consult the Puri & Sharma, Physical Chemistry).
3. Learn the terms: neutralization, salt hydrolysis, weak & strong electrolytes, pH.
4. Write the reactions involved in the titrations.
5. Learn the theory behind your calculations.
6. Calculate equivalent weights of NaOH, Na_2CO_3, HCl, H_2SO_4.
7. Calculate pH of following aqueous solutions at 25°C:

   0.1N HCl, 0.1N H_2SO_4

   0.1M HCl, 0.1M H_2SO_4

   0.1M NaOH, 0.1M KOH

   0.1M NaCl, and pure $H_2O(l)$
8. Write the formulas of various species in the reaction mixture corresponding to each end point.
9. Distinguish between end point and equivalence point.

EXPERIMENT NO 2

Object

To Standardize EDTA and determine the temporary and permanent hardness of a given water sample by complexometric titration using EDTA.

Principle

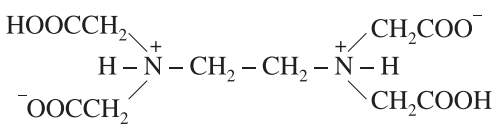
The hardness of water is generally determined by EDTA method. EDTA is a well known complexing agent. It is used as disodium salt of EDTA. Indicator used is metallochromic indicator, they form compounds which is a less stable complex with the metal ions than the metal EDTA complex as a result, the indicator releases the metal ion for complex formation when EDTA solution is added.

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

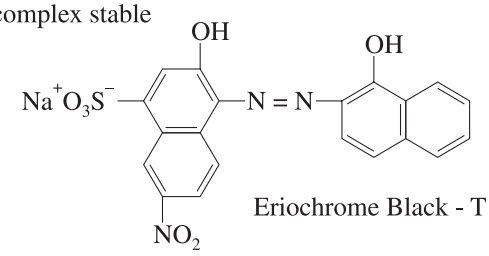
Wine red less stable complex of metal ion + EBT

Colourless metal ion + EDTA complex stable

Blue EBT



EDTA
Six coordination sites
(4 acidic hydrogens and two N-atoms)



Eriochrome Black - T

[Sodium 1-(1-hydroxy-2 naphthylazo)-6 nitro - 2 - naphtol - 4 - sulphanate (1)]

Procedure

(i) **Standardization of EDTA:** EDTA sample is standardized by titrating it against standard hard water sample.

(ii) **Determination of hardness:** Take 10 ml of hard water sample in a conical flask with help of a pipette. Add ¼ test tube of buffer solution. Add 2, 3 drops of Eriochrome black-T indicator. Color of the solution turns wine red. Titrate the solution against EDTA until the color changes from wine red to blue. This reading corresponds to total hardness. Next, take another 10ml. of the hard water in 250 ml beaker with the help of a pipette and boil it for 10 minutes to remove temporary hardness. Cool it and titrate the solution against the same EDTA solution. This end point will correspond to permanent hardness.

Observation

Standardization of EDTA

S.No	Vol of SHW	Vol of EDTA

(1) Total Hardness

(2)

S.No.	Volume of hard water	Titre vol. of EDTA (in burette)
1.	10 ml	
2.	10 ml	
3.	10 ml	

(3) Permanent Hardness

(4)

S.No.	Volume of hard water	Titre vol. of EDTA (in burette)
1.	10 ml	
2.	10 ml	

Result:

Total Hardness = _____ ppm

Permanent Hardness = _____ ppm

Temporary hardness = _____ ppm

Exercise

1. $CaCO_3$ means Ca^{++} solution prepared by dissolving $CaCO_3$ in dil. HCl and its dilution to desired concentration.
2. Note the following colours:

SUBSTANCE	COLOUR
Eriochrome Black-T (EBT)	blue
Ca-EBT Complex	wine red
Mg-EBT Complex	wine red

Ca-EDTA Complex	colorless
Mg-EDTA Complex	colorless

3. Write the structures of EDTA and EBT
4. Write the contents of reaction mixture at the end point.

EXPERIMENT NO 3

Object

To estimate iodine in the given sample of common salt.

Principle

Iodine in common salt exists as NaI. The salt on treatment with glacial acetic acid liberates iodine which is titrated against $Na_2S_2O_3$ solution using starch as a indicator.

$$I_2 + 2Na_2S_2O_3 \rightarrow 2NaI + Na_2S_4O_6$$

(Sodium thiosulphate) (Sodium Tetra thionate)

$$\text{Starch} + I_2 \rightarrow \text{Starch Iodine complex (Blue)}$$

Procedure

Weigh 20g common salt. Transfer it into 250ml. conical flask. Dissolve the salt with minimum amount of water (about 100ml.). Add about half test tube of glacial acetic acid. Iodine will be liberated. Titrate the liberated iodine against N/200 $Na_2S_2O_3$ solution using starch as indicator. (Add the indicator when you are close to the end point). Indicator gives blue color with iodine. 'Just disappearance' of blue color indicates the end point.

Observation

S. No.	Vol. of salt (V_1)	Vol. of N/100 $Na_2S_2O_3$ sol.(V_2)
1	50 ml	
2	50 ml	

Result

The amount of iodine in the given common salt sample is = _____%.

Exercise

1. Learn the terms: Iodometry and Iodimetry.
2. Write the reaction involved during the titration.
3. Calculate equivalent weights of I_2 and $Na_2S_2O_3.5H_2O$.
4. What is the blue species in titration?

EXPERIMENT NO 4

Object

Determine the percentage of available Chlorine in Bleaching powder sample.

Theory

Bleaching powder is used as bleaching agent and also as disinfectant. The main constituent of bleaching powder Calcium hypochlorite supplies Chlorine with dil. Acids as under

$$2CaOCl_2 + 8\ HCl \rightarrow 2CaCl_2 + 4H_2O + \underset{\text{(Available Chlorine)}}{4Cl_2}$$

So, available chlorine is amount of chlorine liberated by the action of dil acids on hypochlorite. It is expressed as percentage by weight of bleaching powder. In principle, the determination of available chlorine is done by treating hypochlorite solution with an excess of a solution of KI, The liberated chlorine reacts with KI solution giving free iodine. This free iodine is then titrated with the standard sodium thiosulphate solution.

$$CaOCl_2 + 2CH_3COOH \rightarrow (CH_3COO)_2Ca + H_2O + Cl_2\uparrow$$

$$KI + Cl_2 \rightarrow KCl + I_2$$

$$Na_2S_2O_3 + I_2 \rightarrow 2NaI + Na_2S_4O_6$$

Chemicals/Reagents

Sample of bleaching powder, KI, dil CH_3COOH (5%) N/10 Sodium thiosulphate.

Indicators

Freshly prepared starch solution.

Procedure

Weigh accurately 2 g of bleaching powder sample in weighing bottle. Transfer it to a porcelain mortar and weigh the bottle again. Difference in weight will give exact weight of the sample taken. Put some distill water on the sample and make a paste. Add more water and grind it, keep it undisturbed for a while and decant milky solution in 250 ml measuring flask. Grind the residue, add more water and repeat the same process until whole of the sample is transferred to the flask. Make up the volume to the mark by adding distilled water and shake it well.

Now pipette out 10 ml of the above solution to a titration flask. Add 2 ml of KI solution and 2 ml dil CH_3COOH. To this add 2-3 drops of freshly prepared starch as indicator, titrate the librated I_2 with standard hypo solution until the disappearance of blue color. Repeat to get concordant reading.

Observation Table

S.N.	Vol of bleaching powder	Vol of $Na_2S_2O_3$

Calculation

1. Normality of bleaching powder

$$N_1V_1 = N_2V_2$$

2. Amount of chlorine in 1L of solution = N_1 × equivalent weight of chlorine = Bg
3. Amount of chlorine present in 250ml of the solution

$$= B/100 \times 250g = Cg$$

4. % of available chlorine = C/W × 100%

Result

The percentage of available Chlorine in Bleaching powder sample is .....

EXPERIMENT NO 5

Object

(i) Determine the strength of $AgNO_3$ solution being provided with N/50 NaCl solution.

(ii) To determine the chloride content in given water sample by Mohr's method.

Principle

Mohr's method is used to determine chloride content in a water sample. In this method, chloride ion solution is titrated against standard silver nitrate solution using potassium chromate as indicator. $AgNO_3$ is taken in burette. The chloride ion solution is taken in pipette, and transferred into a conical flask. To the latter solution K_2CrO_4 indicator is added. As the titration proceeds, the chloride ions present in the solution react with $AgNO_3$ forming insoluble white precipitate of AgCl. At the end point all chloride ions are completely precipitated. The extra drop of $AgNO_3$ from the burette reacts with sodium chromate (indicator) forming reddish brown silver chromate. The appearance of distinct reddish brown precipitate over the white precipitate marks the end point.

$$NaCl + AgNO_3 \rightarrow \underset{\text{White ppt.}}{AgCl} + NaNO_3$$

$$2AgNO_3 + K_2CrO_4 \rightarrow \underset{\text{Reddish brown}}{Ag_2CrO_4} + 2KNO_3$$

Procedure

Pipette out 10 ml of water sample in a conical flask. Add 2-3 drops of freshly prepared K_2CrO_4 solution. Titrate it against standard $AgNO_3$ solution until the reddish brown color persists. Repeat the titration till two concordant readings are obtained.

Observation

Standardization of AgCl

S.No	Vol of N/50NaCl	Vol of AgCl

S.No.	Vol. of water sample (V_1)	Vol. of $AgNO_3$ sol. (V_2)
1	10 ml	
2	10 ml	
3	10 ml	

Result

The amount of chloride ions present in given water sample = ...

Exercise

1. Calculate solubilities of AgCl and Ag_2CrO_4. Given, Ksp values of AgCl and Ag_2CrO_4 are 1.6×10^{-10} and 9.0×10^{-12} respectively at 25°C.
2. Calculate [Ag+] in saturated solutions of AgCl and Ag_2CrO_4 at 25°C.
3. Why does AgCl precipitate before Ag_2CrO_4 during the titration?
4. What are the colours of AgCl and Ag_2CrO_4?

EXPERIMENT NO 6(A)

Object

To determine the iron content in the given sample by redox titration using external indicator.

Theory

Potassium dichromate in acid medium oxidizes ferrous ions present in Mohr's salt into ferric ions. In this titration, potassium ferricyanide is used as an external indicator which gives a blue colour due to the formation of ferro ferricyanide.

$$Cr_2O_7^{2-} + 14H^+ + 6e^- \rightarrow 2Cr^{3+} + 7H_2O$$
$$Fe^{2+} \rightarrow Fe^{3+} + e^- \quad X6$$
$$6Fe^{2+} + Cr_2O_7^{2-} + 14H^+ \rightarrow 6Fe^{3+} + 2Cr^{3+} + 7H_2O$$
$$3Fe^{2+} + 2[Fe(CN)_6]^{3-} \rightarrow 2Fe_3[Fe(CN)_6]$$

Procedure

(i) **Standardization of potassium dichromate by N/20sodium thiosulphate:** Take Sodium thiosulphate in burette. Take potassium dichromate in conical flask and add sulphuric acid, KI and sodium bicarbonate and starch as an indicator, solution turns blue. When titrated against $Na_2S_2O_4$ blue color disappears.

(ii) **Determination of iron content**: Pipette out 10 ml of the sample solution in a conical flask and Titrate the solution against acidified N/20 $K_2Cr_2O_7$ solution, using potassium ferricyanide as an external indicator. Take 1 drop of the solution from the conical flask and put it over a drop of potassium ferricyanide solution placed on a white glazed tile. If a blue color appears, then the end point has not reached. Add more $K_2Cr_2O_7$ solution till a drop of solution dose not changed to blue. This is the end point. Repeat till three concordant readings are obtained.

Standardization of $K_2Cr_2O_7$

S.No	Vol of N/20 Thiosulphate	Vol of $K_2Cr_2O_7$

Observation

S.No.	Vol. of sample Sol. Takes V1	Titre vol. of N/20 $K_2Cr_2O_7$ Sol. V_2
1	10 ml	
2	10 ml	
3	10 ml	

Result

The iron content in the given sample of iron is ..... ppm.

Exercise

1. Calculate equivalent weights of $K_2Cr_2O_7$ and Mohr's salt.
2. What is a double salt? Write names and formulas of two double salts.
3. Write down the name and formula of the blue complex formed with the indicator.
4. Write the chemical reaction of the Titration.

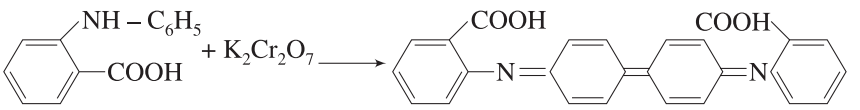
EXPERIMENT NO 6(B)

Object

To determine the iron content in the given sample by redox titration using internal indicator.

Theory

Indicator used is N-phenyl anthranilic acid.



Voilet red

Procedure

Take $K_2Cr_2O_7$ solution in a burette and 10 ml of FAS solution in 250 cm^3 conical flask add ½ test tube of dil H_2SO_4. Then add 4, 5 drops of indicator solution. The $K_2Cr_2O_7$ solution from burette is then slowly added till color changes to violet red.

Observation

S.No.	Volume of sample solution taken V_1	Titre volume of N/20 $K_2Cr_2O_7$ solution V_2
1.		
2.		
3.		

Result

The iron content in the given sample of iron is..... ppm.

EXPERIMENT NO 7

Object

To determine the intrinsic viscosity (η') of a given carbohydrate solution

Principle

Relative viscosity = $\eta_{solution}/\eta'_{solvent} = (d_{solution}/d_{solvent}) \times (t_{solution}/t_{solvent})$

$$\eta_{sp} = \frac{\eta_{solution} - \eta_{solvent}}{\eta_{solvent}} = \frac{\eta_{solution}}{\eta_{solvent}} - 1$$

Intrinsic viscosity = $[\eta]' = \underset{e \to 0}{\text{Lim}} (\eta_{sp}/C)$

Procedure

Weigh 20.00 g sucrose accurately. Transfer it into 100 ml volumetric flask. Dissolve in distilled water and make up to the mark with distilled water. This is 20% sucrose solution (stock solution). Prepare 15%, 10%, 5% and 1% sucrose solutions and find viscosities. Find relative densities of solutions with density bottle.

Observation

S No.	Concentrate of sugar sol.	$\frac{d_{solution}}{d_{water}}$	Flow time	Relative Viscosity	Specific Viscosity	Specific Viscosity / Concentration

CALCULATION

Result

The intrinsic viscosity (η') of a given sugar solution is ..... centipoise.

Exercise

1. Define relative viscosity, specific viscosity and intrinsic viscosity (η').
2. What is meant by laminar flow of a liquid?
3. What is the practical unit of Viscosity?
4. How is molecular weight related to viscosity?
5. How is viscosity related to molecular interaction?
6. Why is viscosity of water lower than that of glycerol?
7. What is meant by relative density?

EXPERIMENT NO 8

Object

Determination of concentration of NaOH solution by titrating it against standard HCl solution using a pH-meter.

Principle

When an alkali is added to an acid solution, the pH of the solution increases slowly. But in the vicinity of the equivalence point, this change of pH is very rapid. From the sharp break in the curve, we can find the equivalence point.

Procedure

First standardize the pH meter with a buffer of pH 4. Take 10ml of HCl solution in a 400 ml. beaker. Add equivalent amount of water so that the electrodes are completely dipped. Note the pH. Next add NaOH from the burette. Stir the solution after each addition and note the pH of the solution after each addition. Near the equivalence point, the alkali should be added in fractions of 0.2 or 0.1 ml. as here, the rise in pH is sharp. Standardized the pH meter with buffer of pH 9 when the pH reaches 7.

Observation

Volume of HCl taken = 10 ml

S.No.	Titre vol. of NaOH	pH	S.No.	Titre vol. NaOH	pH

Calculation

$$N_1V_1 = N_2V_2$$

$$\frac{1}{20} \times 10 = N_2 \times x,\ N_2 = y$$

Strength of NaOH = $(y) \times 40 \times 1000$ ppm

Result

The strength of the given NaOH solution is …….. ppm.

Exercise

1. Define pH
2. Calculate pH of water, N/10 HCl, N/10 NaOH, N/10 H_2SO_4, M/10 H_2SO_4, M/10 NaCl at 25°C
3. Why do you observe pH less than 7 at the equivalence point.
4. What is the effect of temperature on pH of water?

EXPERIMENT NO 9

Object

To determine the concentration of $KMnO_4$ solution spectrophotometrically.

Requirements

Calorimeter, Cuvette, Test Tubes, Beakers etc.

Chemicals

Standard solution and unknown solution of $KMnO_4$

Theory

It is based on Beer's law**.** The variation of colors of the system with concentration is the basis of colorimetry. Colorimetric analysis is especially useful for systems in which substances or their solutions are colored. When a substance is colored then a suitable complexing agent is added to the solution so that a colored complex is obtained, the later than absorbs light in the visible region.

Procedure

(i) Fix the wavelength of the spectrophotometer at λ_{max} 540 far $KMnO_4$/adjust filter.
(ii) From the stock solution of 0.001M make $KMnO_4$ solutions of 1.0, 2.0, 3.0, 4 and 5 concentrations respectively.
(iii) Measure the absorbance values of the above solution *s* at $\lambda_{max.}$
(iv) Plot a graph between absorbance (along x-axis) vs. concentration (along y-axis). Fit all the observation points on the straight line. Beer's law is verified, if a straight line is obtained.
(v) Now find the absorbance value of unknown solution also at $\lambda_{max.}$
(vi) From straight line graph of absorbance vs. concentration, find out the concentration of the unknown solution.

Observations

Concentration of $KMnO_4$ (10^{-4}M)	1.0	2.0	3.0	4.0	5.0	Unknown
Absorbance						

Result

The concentration of unknown $KMnO_4$ solution is ..... M

Precautions

1. What is Beer's Law?
2. What is the λ_{max} of $KMno_4$?
3. What is the Lambert's law?

EXPERIMENT NO 10

Object

Detection Sensitivity the elements in given organic compound

Theory

Test for N, S, and halogens elements

S.NO.	Experiment	Observation	Inference
I	**For Nitrogen**		
1.	Take 2 ml of Sodium Extract + 2 ml. of freshly prepared aq. Sol. of $FeSO_4$+ 1-2 drops of NaOH Green precipitate is obtained. Boil the mixtures cool and add sufficient dil. H_2SO_4 and 2-3 drop of $FeCl_3$ solution.	(a) Blue or green color is obtained (b) Appearance of Red blood color	(a) N present (b) N & S present
2.	Take 2 ml of Sodium extract, acidify with CH3COOH add 2-3 drops of 1% solution of benzidine in 50% acetic acid and 1-2 drops of 1% solution of copper sulfate	A blue color or precipitate	Nitrogen present
II	**FOR SULPHUR**		
1.	Take 2 ml of sodium extract and add 1-2 ml of freshly prepared solution of sodium Nitroprusside.	A violet color	Sulphur present
2.	Take 2 ml of sodium extract acidify with acetic acid and add a few drop of lead acetate solution	A black precipitate of lead sulfide	Sulphur present
III	**FOR HALOGEN**		
1.	Take 1 ml of sodium extract + 0.5 ml conc. HNO_3, boil cool and add silver nitrate solution.	A curdy white precipitate soluble easily the ammonium hydroxide and insoluble in HNO_3	Chlorine present
		If gives a yellowish white or yellow precipitate respectively	Iodine present
		If the color is yellow	Bromine is present
2.	Sodium Extract + conc. HNO_3 + CCl_4 + chlorine water. Shake well. Observe the color of CCl_4 layer	Violet color	Iodine Confirmed
		Yellow or brown	Bromine is present

Exercise

1. What do you understand by Lassaigne's Test.?
2. What are elements that can be detected and which sodium salts are formed?
3. Why is Sodium metal kept in kerosene oil? What is the nature of sodium extract?
4. Why is CCl_4 layer colored with liberated bromine or iodine and not the aqueous layer?
5. Write the name structure of purple color appears while adding sodium nitroprusside in sodium extract
6. How do you confirm whether the given organic compound is aliphatic or aromatic?
7. Write the name and structure of Prussian blue while testing the presence of nitrogen in organic compound

CHEMICAL REACTIONS OF EXPERIMENT NO. - 10

Lassaigne's test (Sodium fusion extract)

Nitrogen

$$Na + C + N \longrightarrow NaCN$$

Sulphur

$$2Na + S \longrightarrow Na_2S$$

Nitrogen and Sulphur Both

$$Na + C + N + S \longrightarrow NaSCN$$

Halogens

$$2Na + X_2 \longrightarrow 2NaX \text{ (Where X = Cl, Br, I)}$$

(i) **Chemistry of Nitrogen test**

$$FeSO_4 + 2NaOH \longrightarrow Na_2SO_4 + Fe(OH)_2$$

Dirty Green Color

$$Fe(OH)_2 + H_2SO_4 + 6NaCN \longrightarrow Na_4[Fe(CN)_6] + 2NaOH$$

Sodium ferro cyanide

$$3Na_4[Fe(CN)_6] + 4FeCl_3 \longrightarrow Fe_4[Fe(CN)_6]_3 + 12NaCl$$

Ferri Ferro Cyanide[Prussian Blue]

(ii) **Chemistry of Sulphur test**

(a) $Na_2S + Na_2[Fe(NO)(CN)_5] \longrightarrow Na_3[Fe(O = N - S\text{-}Na)(CN)_5]$

Purple Color

(b) $Na_2S + Pb(CH_3COO)_2 \longrightarrow PbS + 2CH_3COONa$

(Black color)

(iii) **Chemistry of Nitrogen and Sulphur both test**

$$3NaCNS + FeCl_3 \longrightarrow Fe(CNS)_3 + 3NaCl$$

Ferric Sulpho cyanide (Red color)

(iv) **Chemistry of Halogens**

$$NaCN + HNO_3 \longrightarrow NaNO_3 + HCN$$

$$NaX + AgNO_3 \longrightarrow AgX + NaNO_3$$

white color ppt, soluble in ammonium hydroxide (Cl present)

$$NaCl + AgNO_3 \longrightarrow AgCl \text{ (white color)}$$

$$NaI + AgNO3 \longrightarrow AgI$$

Yellowish white color ppt, insoluble in ammonium hydroxide

(b) $2NaI + Cl_2 \longrightarrow 2NACl + I_2$ (Voilet color in CCl_4)

$2NaBr + Cl_2 \longrightarrow 2NaCl + Br_2$ (brown or yellow color in CCl_4)

EXPERIMENT NO 11

Object

Identify the Functional Group in given organic compound.

Apparatus used

Beakers, test tubes, tong, distilled water, etc.

Chemicals used

Sodium bicarbonate solution, Neutral Ferric chloride solution, Sulphuric Acid, Sodium Hydroxide, Phthalic anhydride, Sodium Metal, Na_2SO_4, Ethanol, Sodium nitroprusside, etc.

Theory

Test for Functional Groups:

S.No.	Experiment	Observation	Inference
I	**For Carboxylic Acid**		
	Add a pinch of the substance of solid (or a few drops its liquid) +5 ml of saturated $NaHCO_3$ sol. in water	Effervescence due to the evolution of CO_2	–COOH
II	**For Phenols**		
1.	To one of aqueous solution or alcoholic solution of O.S. add 2-3 drops of aq. $FeCl_3$ solution.	Violet, color indicate the presence of phenolic OH group	Violet phenol O– –Violet Resorcinol
2.	Take 0.2 g each of O.S. and phthalic anhydride in a dry test tube, add 0.5 ml of conc. H_2SO_4 and heat. Pour the content of the test tube into 50 ml water containing about 1 ml dil aq. NaOH.	A green fluorescence	Phenol present.
III	**For Carbohydrate**		
1.	Take O.S. in a dry test tube and 1-2 drops of conc. H_2SO_4	A red or crimson color	presence of carbohydrate
2.	Heat 0.2 g of O.S. with 1 ml of conc. H_2SO_4	An immediate charring	presence of carbohydrate
3.	Take 2 ml of aqueous sol. add 1 ml of Molisch reagent and then add carefully 1 ml of conc. H_2SO_4 from the side of the test tube.	A reddish violet ring at the junction of the two liquid is formed.	presence of carbohydrate
IV	**For Ketones**		
1.	Take 2 ml of aqueous or alcoholic solution of O.S., and 0.5 1 ml. of Sodium nitroprusside and 2-3 drops of aq. NaOH	A yellow or red color	presence of a Ketone

2.	Take 1 ml alcoholic solution of O.S. and 1 ml of 2; 4-dinitro phenyl hydrazine solution, boil and cool	A yellow orange or red precipitate	presence of a Ketone
V	**For Aldehyde**		
1.	Take 1 ml of a mixture of equal amounts of Fehling solution A & B, and add a few drops of 2 g of O.S. and heat on a water bath for 5 minutes.	A red precipitate of Cu2O is obtained	presence of an aldehyde
2.	Take 1 ml of Tollen's reagent and a few drops on 0.2 g of O.S. and heat on a water bath for 5-10 minutes.	A silver mirror is obtained due to deposition of metallic silver on the side of the test tube.	presence of an aldehyde.
VI	**For Ester**		
1.	Take compound on watch glass and carefully smell	Pleasant and fruity aroma	Ester is possible
2.	Dissolve one gm of compound in ethanol, add 2-3 drops of dil NaOH solution and add 1drop phenolphthalein reagent, heat for 5 min	Pink color appears	Ester is present
	For amine		
1.	Take one gm of compound + potassium dichromate + conc.sulphuric acid	Red/violet color changes green on heating	amine
2.	Take 1gm of compound and add alcoholic KOH and add 2-3 drops of $CHCl_3$+heat	Intolerable offensive smell	Aliphatic or aromatic amine.
	For alcohol		
1.	Add 1-3 drops of ceric ammonium nitrate reagent in about 1ml of given organic compound	Pink or red color	Alcohol is present
2.	Dissolve unknown compound in acetone and add chromic acid	Within seconds orange disappears and formation of an opaque blue-green suspension takes place	Alcohol present
	Nitro compound		
	Take one gm of compound+ 2ml of ethanol + 1ml of ammonium chloride + pinch of Zn dust + boil and cool and filter into test tube containing Tollen's reagent	Black ppt	Nitro compound
	For Unsaturation		
1.	Dissolve 40 gm/1ml of unknown compound in water or 95% ethanol in a test tube. Drop wise add aq.$KMnO_4$ until purple color persist or shake well and let it stand	Decolorization of permanganate accompanied by formation of reddish brown suspension of MnO_2	Unsaturation is present

Exercise

1. How are phenolic groups identified?
2. Which one is more acidic phenol or carboxylic acid?
3. Learn Molisch's test, Tollen's regent test and Fehling's test
4. Write the chemistry of Phthalien test?
5. Write the reaction when phenol reacts with neutral $FeCl_3$ solution

CHEMICAL REACTIONS OF EXPERIMENT NO. – 11

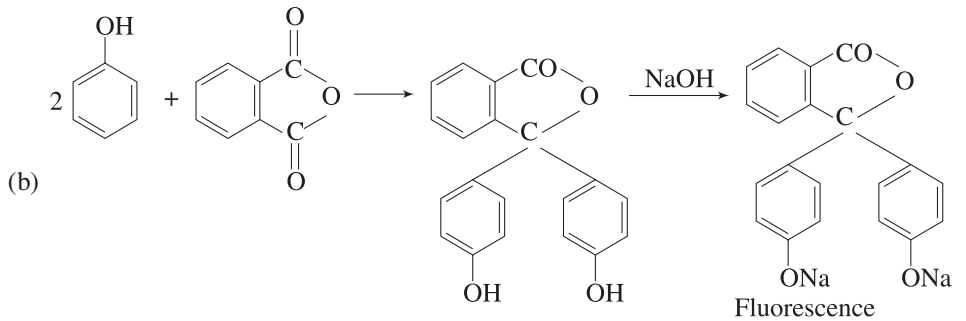
Chemistry of Reactions

(i) $CH_3COOH + NaHCO_3 \longrightarrow CH_3COONa + H_2O + CO_2 \uparrow$

(ii) (a) $6C_6H_5OH + FeCl_3 \longrightarrow 3H^+[Fe(OC_6H_5)_6]^{-3} + 3HCl$

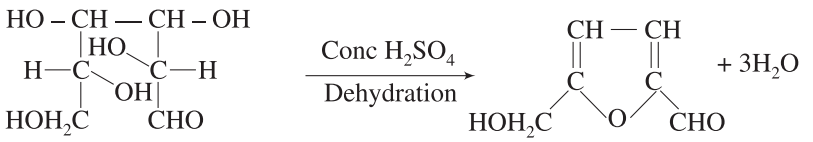
Coloured complexion

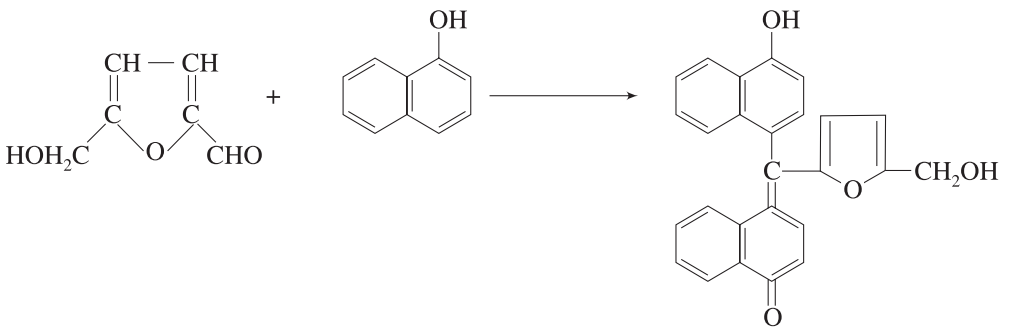
(b)



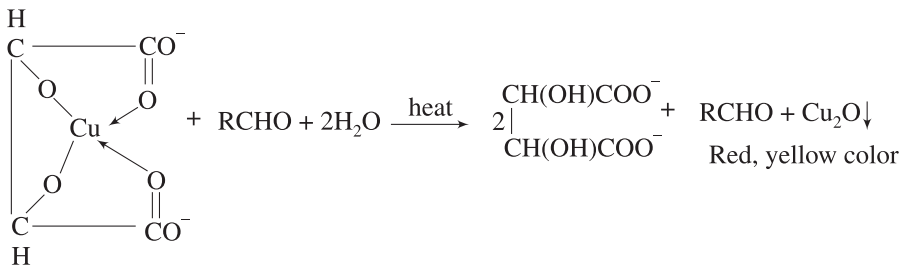
Fluorescence green color

(iii)





(iii) (a)



Red, yellow color

Tartrate - Cu(II)anion

Fehling's solution (Blue)

(b) $RCHO + 2[Ag(NH_3)_2]^+ OH^- \longrightarrow 2Ag\downarrow + RCOONH_4 + H_2O + 3NH_3\uparrow$

Tollen's regent

EXPERIMENT NO 12(A)

OBJECT

To prepare phenol formaldehyde resin (Bakelite).

Requirements

Beaker, measuring cylinder, weight box, chemical balance, filter paper.

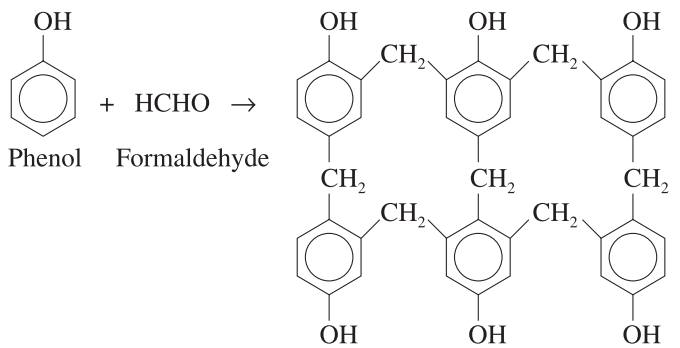
Chemicals

Formaldehyde, glacial acetic acid, phenol, conc. HCl and distilled water.

Theory

Bakelite is a thermosetting plastic; It can be prepared by phenol and formaldehyde in the presence of acidic medium. Bakelite is a hard pink color material which cannot be remolded at high temperature.

Chemical Reactions



Procedure

Place 5 ml of glacial acetic acid and 2.5 ml of formaldehyde solution in a 500 ml beaker and add 2 gms of phenol. Wrap a cloth loosely around the beaker. Then add conc. drop wise into mixture carefully. Keep the above mixture for 5 mins, firstly it will form white ppt., later on pink color hard mass is obtained. Wash the hard mass with distilled water to remove excess of impurities then remove the hard mass into the watch glass, dry and weigh.

Result

Pink material is obtained i.e. Bakelite resin

Exercise

1. What are polymers?
2. What are thermoplastics and thermosetting polymers?
3. Give the monomers of Bakelite.
4. Give the structure of Bakelite.

EXPERIMENT NO. 12(B)

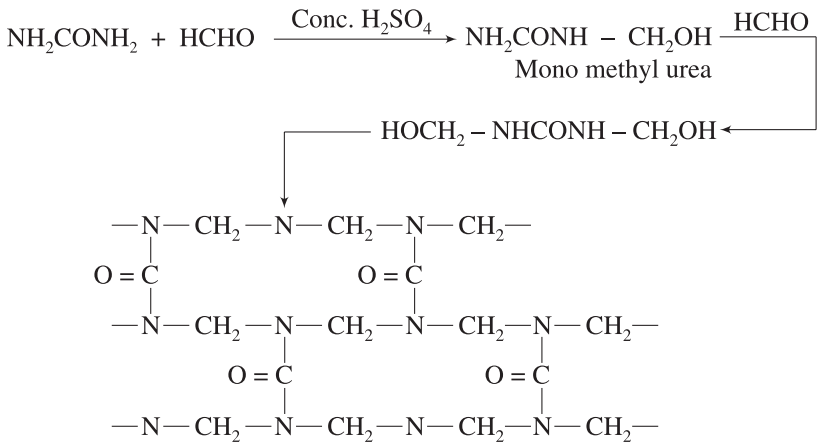
Object

To prepare Urea – Formaldehyde resin.

Chemical Used

Urea, Formaldehyde, conc. H_2SO_4, Porcelain dish, Glass rod

Reaction



Procedure

Take 5 ml of 40% formaldehyde solution in a porcelain dish and add about 5 gm of urea while stirring until a saturated solution is obtained. Add few drops of conc. H_2SO_4 stir continuously during the addition. All of sudden white solid mass appears in the porcelain dish.

Result

white color mass is formed

EXPERIMENT NO 13

Object

Preparation of Alum (K_2SO_4, $Al_2(SO_4)_3$. $24H_2O$).

Requirement

K_2SO_4, $Al_2(SO_4)_3$, dilH_2SO_4, water, beaker, china dish, funnel,
glass rod, tripod stand, wire gauze, burner, wash bottle, jar and balance.

Procedure

1. Take 2.5 gm K_2SO_4 in a beaker.
2. To this add 20ml of distilled water and stir using glass rod using a glass rod until the crystals completely dissolve.
3. Take 10 gm $Al_2(SO_4)_3$ in another beaker.
4. Add 20 ml distilled water and 1 ml dil H_2SO_4.
5. Heat the content for 5 mins
6. Mix the content in two china dish.
7. Heat the content in china dish for sometimes to concentrate it to crystallization point.
8. Tranfer the solution into a crystallizing dish and do not disturb.
9. On cooling crystals of potash alum separate.
10. Decent the mother liquor and wash he crystals with cold water.
11. Dry the crystals by placing between filter paper.
12. Weight the alum, note the color and shape.

Exercise

1. Is alum a complex or a double salt?
2. What are the uses of alum?
3. Give the formula of alum.
4. Give the color and shape of crystal.

EXPERIMENT NO 14

Object

To determine the surface tension of the given liquid at room temperature by stalagmometer.

Materials

Stalagmometer, specific gravity bottle or pyknometer, unknown liquid, rubber tube with screw pinch cock.

Theory

The surface tension of the liquid is determined relative to water at the room temperature by using stalagmometer. The no. of drops for the same volume of water and the given liquid are counted and let these be as n_1 and n_2 respectively. Now if d_1 and d_2 are densities of water and the given liquid at the room temperature as determined separately by specific gravity bottle or pyknometer, then the surface tension y_2 of the given liquid can be calculated by using simplified relationship a

Procedure

1. Clean the Stalagmometer.
2. By immersing lower end in a beaker containing distilled water, suck up water until it rises above the mark C and tighten the screw pinch.
3. Now loosen the screw of the screw pinch carefully so that the liquid drops start falling at an interval of about 2-3 seconds in successive drops. Counting of drops is started when the water meniscus just reaches the upper mark C, and stopped when the meniscus just appears lower mark D. Repeat to get three readings and take the mean value.
4. Clean the Stalagmometer and dry it. Fill it with liquid until it rises above upper the mark C and count the number of drops as before.
5. Measure the density of liquid by specific gravity bottle or pyknometer.

Observations and Calculations:

Room Temp = ... °C

Liquid	**No of Drops**		
	1	2	3
Water			
Given Liquid			

Result

The surface tension of the given liquid

Exercise

1. Define the term Surface Tension?
2. Give the CGS and SI Units of Surface Tension?
3. Why liquids rise in capillary tube?

EXPERIMENT NO 15

Object

Separate red ink and blue ink from a mixture by paper chromatography.

Theory

Chromatography is defined as separation of molecular mixtures by distribution between two or more phases, one phase essentially be two dimensional and the remaining being bulk phase brought into contact in a counter-current fashion with the two dimensional phase. It is based upon preferential absorption of a substance on particular absorbent surface.

Requirement

Strip (2.5*15cm) of whatmann no. 1, wide bore test tube, corks cut vertically into halves, solvent (*n*-butanol : acetic acid : water in ratio of 4:1:5) red ink, blue ink, capillary tubes.

Procedure

1. Take filter strip and mark a pencil line at 1cm from bottom edge and 3 cm from top edge. Apply mixture of blue and red ink with the help of capillary at centre of the bottom pencil line. Dry the spot.
2. Add about 5 ml of solvent in wide bore test tube. Suspend the filter paper strip into wide test tube so that lower end dips in the solvent taking care hat spot does not touch the solvent.
3. Keep the test tube undisturbed for nearly 2-3 hrs until the solvent rises two a level marked with pencil. Now remove and dry the strip. The filter strip is now know as chromatogram. Draw a pencil on each chromatogram at the level to which the solvent has risen.

   Determination of *Rf* value On each chromatgram measure the distance moved by spot and the distance moved by the solvent. Calculate the value of Rf from the relation-

   Rf = distance move by solute/distance moved by solvent

Exercise

1. Give the basic principle of chromatographic techniques.
2. Can absolute value of Rf value be assigned?
3. On what factors does absorption chromatography depends?
4. On what factors adsorption chromatography depends?