RPS DEGREE COLLEGE

BALANA (MAHENDERGARH)-123029

Lab Manual

Chemistry (B.Sc.1st & 2nd Semester)

Department of Chemistry

INORGANIC CHEMISTRY

A. Gravimetric Analysis
1. Quantitative estimations of Cu^{+2} as copper thiocyanate
2. Quantitative estimations of Ni^{+2} as Ni-dimethyl glyoxime.

B. Colorimetry
3. To verify Beer lambert law for $KMnO_4/K_2Cr_2O_7$ and determine the concentration of given $KMnO_4/K_2Cr_2O_7$ solution .

C. Preparations
4. Preparation of cuprous chloride
5. Preparation of Prussian blue from iron fillings
6. Preparation of tetraamine cupric sulphate
7. Preparation of chrome alum
8. Preparation of potassium trioxalatochromate(III)

PHYSICAL CHEMISTRY

9. To determine the enthalpy of solution of solid calcium chloride
10. To study the distribution of iodine between water and CCl_4
11. To determine the CST of phenol water system
12. To determine the solubility of benzoic acid at various temperatures and to determine the ΔH of the dissolution process
13. To determine the enthalpy of neutralization of a weak acid /weak base vs. strong base/strong acid and determine the enthalpy of ionization of weak acid/weak base

ORGANIC CHEMISTRY

14. To analyze the given organic compound in a systematic way i.e. detection of extra element ,functional group , determination of melting point , preparation of solid derivatives of following compounds : Naphthalene, oxalic acid, β-Napthol ,benzophenone , pthalic acid , aspirin, p-nitro toluene, anthracene ,m-dinitrobenzene , benzyl chloride , glucose, fructose, urea, cinnamic acid , benzamide.

EXPERIMENT 1

AIM : - Find out gravimetrically the percentage of Cu in given solution of $CuSo_4.5H_2O$, 20g of which has been dissolved per litre.
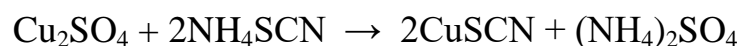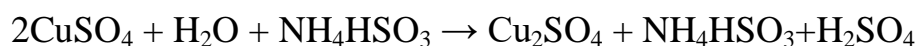
APPARALUS REQUIRED:-
Beaker, watch glass, pipette, glassrod.

CHEMICAL REQUIRED:-
1. Ammonium thiocyanate solution.
2. Saturated solution of NH_4HSO_3.
3. Solution of $CuSO_4.5H_2O$.

REACTIONS:-

$$2CuSO_4 + H_2O + NH_4HSO_3 \rightarrow Cu_2SO_4 + NH_4HSO_3 + H_2SO_4$$

$$Cu_2SO_4 + 2NH_4SCN \rightarrow 2CuSCN + (NH_4)_2SO_4$$

OBJECTIVE:-
In this experiment firstly, cupric salt is reduced to cuprous salt either by using sulphurous acid or ammonium bisulphate solution. Then cuprous ions are precipitated by ammonium thiocyanate (NH_4SCN). After then precipitates formed are filtered, washed dried and weighed using sintered glass crucible. From the weight of precipitates formed percentage of copper is determined.

PROCEDURE:-
1. Take 20 ml of given solution of copper sulphate in a clean 400 ml beaker.
2. Add few drops of HCl, followed by addition of 25 ml of 10% NH_4HSO_3 Solution.
3. Dilute the above solution by adding 150 ml of distilled water. Boil the solution.
4. Now put the beaker on an asbestos sheet, and then add drop wise 10% ammonium thiocyanate solution with constant stirring till the supernatant solution becomes colourless. (To check for complete precipitation add few drops more of ammonium thiocyanate).
5. Cover the beaker and allow the contents to stand for 2-3 hours preferably overnight.

6. Filter the precipitates using G-4 crucible, wash the precipitates using 1% cold dilute solution of NH_4HSO_3 till the filterate is free from SCN^- ions.
7. Finally wash the precipitates with 20% alcohol to remove NH_4SCN.
8. Heat the crucible in oven at 110^0 - 120^0C to constant weight.

OBSERVATIONS:-

Volume of given solution = 20ml

Strength of given Copper sulphate solution=20 g/litre

Weight of sintered glass crucible Before experiment i.e., empty=W_1g

Weight of sintered glass crucible
and $Cu_2(SCN)_2 = W_2$g

Weight of $Cu_2 (SCN)_2$ formed = $w_{2-W}1$ =Wg

CALCULATIONS:-

I.   From 20 ml of given solution weight $Cu_2(SCN)_2$ formed = Wg

$$\text{From 1 ml of given solution weight} = \frac{W}{20}$$

From 1000 ml given solution weight = $\frac{W}{20} \times 1000 = 50$ Wgm

II. $Cu_2(SCN)_2$ $=$ 2 cu

243 gm $Cu_2(SCN)_2$ is formed from copper =127 gm

1 gm $Cu_2(SCN)_2$ is formed from copper $= \frac{127}{243}$

50w gm of $Cu_2(SCN)_2$ is formed from copper =

$\frac{127}{243} \times 50\,w = a\,gm-litre\,(say)$

III.   In 20.0n gms of copper sulphate, actual amount of Cu present = a gms

In 1 gms of copper sulphate,

$$\text{Actual amount of Cu present } = \frac{a}{20}$$

In 100 gms of copper sulphate,

$$\text{Actual amount of Cu present} + \frac{a}{20} \times 100 + 5a$$

RESULT:-

Percentage of Cu = 5a

PRECAUTIONS:-

1. Precipitate i.e. NH_4SCN should not added in excess to avoid solubility of $Cu_2(SCN_2$ as complex ion
2. Washing of the precipitates is to be done with dilute solution of NH_4HSO_3 to avoid oxidation of Cu(I) to Cu (II)

EXPERIMENT:-2

AIM :- Find out gravimetrically the percentage purity of $NiSO_4. 7H_2O$, 20.0g of which has been dissolved per litre.

CHEMICAL REQUIRED:-
1. One % dimethyl glyoxime solution in alcohol
2. Aqueous ammonia solution
3. Solution of nickel salt

APPARATUS REQUIRED:-
Pipette, glass rod, watch glass, beaker, sintered glass crucible

PROCEDURE:-
1. Take a 400 ml beaker add into it 20 ml of given nickel salt solution with the help of pipette.
2. Add 5 ml of HCl and dilute the mixture by adding nearly 200 ml distilled water. Heat the solution to 70 – 80℃
3. Place the beaker on an asbestos sheet and add slowly 30 ml of 1% alcoholic DMG solution with constant stirring.
4. After it adds immediately ammonia solution with constant stirring till it gives ammonical smell.
5. Now keep the beaker undisturbed for 45 minutes approx. Then digest the precipitates on a water bath for 30 minutes.
6. Cool the solution and check for complete precipitation by adding two drops of DMG.
7. Now filter the precipitates using G-3 crucible using a suction pump.
8. Wash the precipitates with small lots cold water several times.
9. Dry the crucible containing precipitates in an electric oven to constant weight.

OBSERVATION:-

Vol. of given solution taken for precipitation = 20 ml

Strength of given nickel sulphate solution = 20.0 gms/litre
Weight of sintered glass crucible before experiment i.e., empty = W_1 gms
Weight of sintered glass crucible and nickel dimethyl glyoxime = W_2 gms
Weight nickel dimethyl glyoxime formed = $W_2 – W_1$ = W gms
CALCULATIONS:-

I. From 20 ml of given solution weight of nickel dimethyl glyoxime formed = W gms

From 1 ml of given solution weight of nickel dimethyl glyoxime formed $= \frac{W}{20}$

From 1000 ml of given solution weight of nickel dimethyl glyoxime formed $= \frac{W}{20} \times 1000 = 50\, W\ gms$

II. $Ni(C_4H_7O_2N_2)_2 = NiSO_4 \cdot 7H_2O$

288.7 gms of nickel dimethyl glyoxime is obtained from $NiSO_4 \cdot 7H_2O = 280.7$ gms

1 gm of nickel dimethyl glyoxime is obtained from $NiSO_4 \cdot 7H_2O = \frac{280.7}{288.7}$

50 w gms of nickel dimethyl glyoxime is obtained from $NiSO_4 \cdot 7H_2O$

$$= \frac{280.7}{288.7} \times 50\ W = a \text{ g/litre (say)}$$

III. In 20.0 gms of impure sample of $NiSO_4 \cdot 7H_2O$, actual amount of $NiSO_4 \cdot 7H_2O$ present = a g

In 1 gm of impure sample of $NiSO_4 \cdot 7H_2O$ actual amount of $NiSO_4 \cdot 7H_2O$ present $= \frac{a}{20}$

In 100gms of impure sample of $NiSO_4 \cdot 7H_2O$ present $= \frac{a}{20} \times 100 = 5a$

RESULT:-

Percentage of purity of $NiSO_4 \cdot 7H_2O$

Sample = 5a%

PRECAUTIONS:-

1. Excess addition of precipitant is avoided, so that precipitant may not get separated along with the precipitates.
2. To avoid decomposition of precipitates the drying should not carried out above 120℃.

Experiment:3

Aim:-To verify Beer-Lambert law for KMnO4 and determine the concentration of the
given KMnO4 solution.

Chemical Required:- solid KMn04.

Apparatus Required:- Spectrophotometer or Elico colorimeter, measuring flasks (100ml and 1000ml), weight box, fractional weights, graph papers.

OBJECTIVE:-

In it we used Beer Lamberts law, this law was dependent on absorbance phenomena.For it number of standard solutions of different concentrations are prepared. Their absorbance is determined. Then a plot of A vs c is drawn. It is a straight line passing through the origin. This proves the validity of Beer-Lambert law. Then the absorbance of the unknown solution is determined under the same experimental conditions. The concentration corresponding to this absorbance is read from the calibration graph.

PROCEDURE:-

(i) Prepare a stock solution of 10^{-3}M KMn04 by dissolving 0.0316g solid KMn04 in one liter distilled water.

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(ii) Took four 100ml flat-bottomed measuring flasks and name them as A, B, C and D respectively. (iii)Now pipette out 20, 40, 60 and 80ml of stock solution of KMn04 into flask A, B, C and D respectively. Make the solution up to the given mark in Conical flask by dilution with distilled water in every 100ml flask.

20ml stock solution+   40ml stock sol.+   60ml stock sol+   80ml stock sol+

80ml distilled   60ml water   40ml water

20ml water

<center>**A**   **B**   **C**</center>
<center>**D**</center>

(iv) Fill one optical cell with the stock solution and 2nd with distilled water. Insert them in colorimeter. Insert different filters one by one and find the filter that gives maximum absorbance. This filter is not to be changed throughout the experiment.

(v) Remove the stock solution and fill that optical cell with the solution (minimum 4ml) from flask A. Note the absorbance. Repeat the experiment with solution from flask B, C and D and note the absorbance in each case. Plot the calibration curve between *A* vs c.

(vi) Now fill the unknown solution and note the absorbance.

OBSERVATION AND CALCULATION:-

Solution	Concentration	Absorbance
Stock solution	10^{-3} M	....
Flask A	0.2×10^{-3} M	....
Flask B	10^{-3} M	....
Flask C	0.6×10^{-3} M	....
Flask D	10^{-3} M	....

From the calibration curve, read the concentration of the unknown solution corresponding to the absorbance. Further a straight line verified the Beer-Lambert's law.

RESULT:

The concentration of given KMnO4 solution is………………… ....

Calibration curve
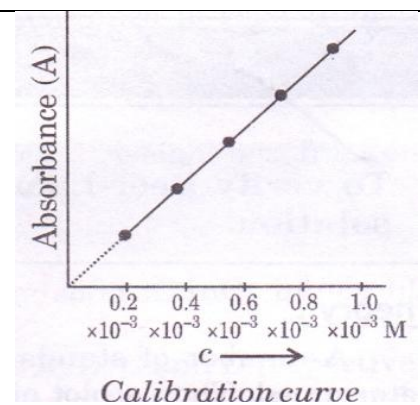
EXPERIMENT NO. 4

AIM :- Prepare a pure sample of cuprous schloride, Cu_2Cl_2

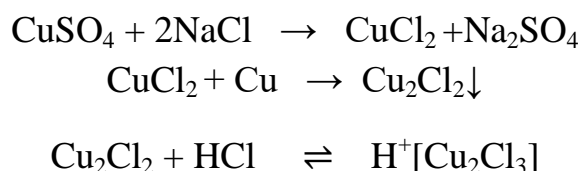**APPARATUS REQUIRED** :- Tripod stand, burner, funnel, round bottom flask, measuring cyclinder, etc.

CHEMICAL REQUIRED:-
1. Copper turnings = 10 gm
2. Sodium Chloride (NaCl) = 5 gm
3. Sodium sulphite = 5 gm
4. Copper sulphate ($CuSO_4. 5H_2O$) = 10gm
5. Conc. HCl = 30ml

CHEMICAL REACTION:-

$$CuSO_4 + 2NaCl \rightarrow CuCl_2 + Na_2SO_4$$
$$CuCl_2 + Cu \rightarrow Cu_2Cl_2\downarrow$$

$$Cu_2Cl_2 + HCl \rightleftharpoons H^+[Cu_2Cl_3]$$

OBJECTIVE:- Cuprous chloride (Cu_2Cl_2) can be obtained by action of cupric chloride with excess copper in acidic solutions in presence of NaCl. Mixture is heated till the solution becomes colorless. The Colourless solutions when diluted results in formation of white powder i.e Cuprous chloride.

PROCEDURE:-
1. Measure 10 gm powdered Copper Sulphate, 10 gm Copper turnings, 5 gm Sodium Chloride and 30 ml of conc. HCl in a 250 ml round bottom flask.
2. Now heat the above mixture with occasional shaking. Continue the heating till the blue colour of solution disappears and a straw colour is obtained. It is the end point of heating.
3. Now stop the heating and pour the contents into a beaker containing 300 ml waterd.
4. Filter the white colour ppt. obtained and wash with very dilute solution of sodium sulphite.
5. Finally wash the ppt. with alcohol and ether.
6. Dry the ppt. in oven or by use of filter paper.

RESULT:-
1. Yield = 4 -6 gm
2. Colour = White

PRECUATIONS:-

1. Cuprous Chloride prepared is kept in a properly corked dry test tube to avoid oxidation of copper due to which it changes to green colour.
2. HCl should be added carefully.

EXPERIMENT 5

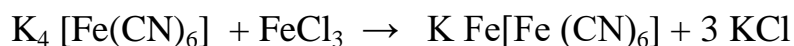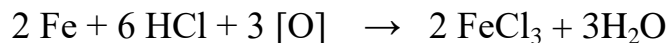**AIM:-** To prepare pure crystals of Prussian blue from Iron fillings.

APPARATUS REQUIRED:- Measuring cyclinder, beaker, dropper, water bath, wire gauze, etc.

CHEMICAL REQUIRED:-
1. Potassium ferrocyanide ($K_4[Fe(CN)_6]$) = 25 gm
2. Iron fillings = 5 gm
3. Conc. HCl = 25 ml
4. Conc. HNO_3 = 5 ml

OBJECTIVE:- Prussian complex can be prepared by adding potassium ferrocyanide to ferric salt. A dark blue coloured compound is obtained i.e. Prussian complex.

REACTIONS:-
$$2\ HNO_3\ (Conc.)\ \rightarrow\ H_2O + 2NO_2 + [O]$$

$$2\ Fe + 6\ HCl + 3\ [O]\ \rightarrow\ 2\ FeCl_3 + 3H_2O$$

$$K_4\ [Fe(CN)_6]\ + FeCl_3\ \rightarrow\ K\ Fe[Fe\ (CN)_6] + 3\ KCl$$
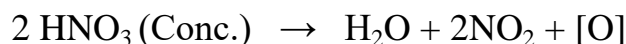
PROCEDURE:-
1. Weigh out 5 gm of Iron fillings in a clean 250 ml beaker. Pour into it 20 ml conc. HCl and 5 ml conc. HNO_3.
2. Allow the above mixture to stand for sometime till the reaction starts.
3. Now heat the contents of the beaker till the fumes are removed.
4. Now add 25 gm of Potassium ferrocyanide with constant stirring till blue colour ppt. formed.
5. Filter the ppt. and wash with dil. HCl.
6. Dry the ppt. in oven or by using filter paper.

OBSERVATIONS:-
1. Colour – blue
2. Yield = 18 to 20 gm

PRECAUTIONS:-

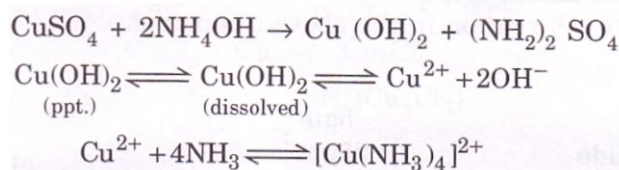1. Pottasium ferrocyanide in powder form should be added to hot solution with constant stirring.

Experiment:6

Aim:-To prepare pure crystals of tetra ammine copper (II) sulphate.

Objective:-_____

Tetra ammine copper sulphate is a complex of Cu(II) with co-ordination no. 4. When NH_4OH i: gradually added to an aqueous solution of$CuSO_4$, $CU(OH)2$ is first precipitated and the precipitate ther dissolves, yielding a solution of a bright blue colour which is due to formation of tetra ammine copper (II) ion, $[Cu(NH_3)4]$

$$CuSO_4 + 2NH_4OH \rightarrow Cu\ (OH)_2 + (NH_2)_2\ SO_4$$
$$\underset{\text{(ppt.)}}{Cu(OH)_2} \rightleftharpoons \underset{\text{(dissolved)}}{Cu(OH)_2} \rightleftharpoons Cu^{2+} + 2OH^-$$
$$Cu^{2+} + 4NH_3 \rightleftharpoons [Cu(NH_3)_4]^{2+}$$

The removal of practically the whole cupric ion from the solution shifts the equilibrium of equation (2) towards the right, until the cupric hydroxide has completely passed into solution. The deep blue solution containing tetraamminecopper (II) ion is known as **Schweitzer's reagent.**
It has an ammonical odour, and crystals ofthe complex are obtained by adding ethyl alcohol to the above solution.

CHEMICAL REQUIREMENTS:-

Copper sulphate	5gm
1 : 1 Ammonia	20ml
Ethyl Alcohol	20-25ml
Conc. H_2SO_4	1-2ml

APPARTUS REQUIRED:- Beaker,Burner,Measuring Cylinder.

PROCEDURE:-

(i) Take 5gm powdered copper sulphate in a 250ml clean beaker and dissolve it **in** a minimum amount of water. Add 1-2ml cone. H_2SO_4 to make the solution clear.

(ii) Now pour 1 : 1 NH_4OH very slowly into the beaker with constant stirring till a ppt. of Cu(OH)2 first formed is redissolved yielding a deep blue solution due to formation of $[Cu(NH_3)4j2+$ and smell of ammonia is present due to slight excess of NH_4OH.

(iii) Now to the blue solution, add 20-25ml of ethanol dropwise with constant stirring. Add about 2ml of NH_4OH and heat the beaker in a water bath at 60-70°C for about 15-20 minutes.

(iv) Stop heating, cover the beaker with clock glass and allow it to stand undisturbed for 2-3 hours   (preferably overnight)

(v) Long needle shaped blue crystals of complex are formed. Filter, wash with little alcohol and dry the crystals gently by pressing in between the folds of the filter paper or by placing in a desiccator.   .

OBSERVATIONS:-

   (i)   Yield= about 5gm

   (ii)   Colour = Blue

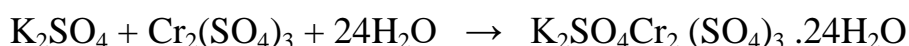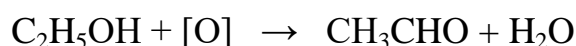   (iii)   Shape = Needle-shaped crystals

EXPERIMENT: 7

AIM:- To prepare pure crystals of chrome alum.

APPARATUS REQUIRED:- Measuring cyclinder, ice bath, round bottom flask etc.

CHEMICAL REQUIRED:-
1. Potassium dichromate ($K_2Cr_2O_7$) = 5 gm
2. Ethyl alcohol (C_2H_5OH) = 5 - 10 ml
3. Conc. H_2SO_4 = 5 ml
4. Water = 20 – 25 ml

REACTIONS :-

$$K_2Cr_2O_7 + 4H_2SO_4 \rightarrow K_2SO_4 + Cr_2(SO_4)_3 + 4H_2O + 3[O]$$

$$C_2H_5OH + [O] \rightarrow CH_3CHO + H_2O$$

$$K_2SO_4 + Cr_2(SO_4)_3 + 24H_2O \rightarrow K_2SO_4Cr_2(SO_4)_3 .24H_2O$$

OBJECTIVE:-

When acidified $K_2Cr_2O_7$ solution is reduced by ethyl alcohol and the reduced solution is allowed to stand dark purple coloured crystals of chrome-alum separates out.

PROCEDURE:-
1. Dissolve 5 gm of $K_2Cr_2O_7$ in about 20 ml of water in round bottom flask and add 5 ml of conc. H_2SO_4 to it.
2. Shake well till whole of solid dissolves.
3. Cool the contents at room temperature in placing ice bath.
4. Now add dropwise ethyl alcohol with constant stirring. take care that the temperature does not exceed 50^0 C. If required add 2 – 3 pieces of ice.
5. Cool the solution in ice bath and allow the contents to stand for about 10 hours.
6. Filter and wash the crystals with cold water and dry it using filter paper. Dark purple colour crystals formed that are chrome alum crystals.

RESULT:-
1. Colour - dark purple
2. Yield = 10-12 gm

PRECAUTIONS:-

1. Add conc. H_2SO_4 carefully.
2. Take care that temperature should not exceed 50^0C.

EXPERIMENT: 8

AIM:- To prepare pure crystals of potassium trioxalatochromate (iii)

APPARATUS REQUIRED:- Stirrer, beaker, measuring cyclinder, ice bath, glass rod etc.

CHEMICAL REQUIRED:-
1. Oxalic acid = 12 – 15 gm
2. Ethyl alcohol = 10 – 15 ml
3. Potassium dichromate = 5 gm
4. Potassium oxalate monohydrate = 6 gm

OBJECTIVE:-
Potassium trioxalatochromate can be obtained by mixing potassium dichromate with oxalic acid and potassium oxalate. The mixture on cooling yields the crystal of the complex.

PROCEDURE:-
1. Add 5 gm of powdered $K_2Cr_2O_7$ in a minimum amount of boiling distilled water in 100 ml beaker.
2. Dissolve 15 gm of powdered oxalic acid in about 25 ml distilled water in a 250 ml beaker separately.
3. Cool the dichromate solution and pour its slowly in small amount to oxalic acid solution.
4. After the completion of reaction the solution is heated to half of the volume of the mixture.
5. Pour 6 gm of potassium oxalate slowly in small amount with constant stirring till the potassium oxalate completely dissolves.
6. Now add 10 – 15 ml ethyl alcohol to the above mixture and place it in ice. Bluish green crystals of the complex separates out.
7. Filter the ppt. and dry them using oven or by filter paper.

RESULT:-
1. Colour – Bluish green
2. Yield = 12 – 13 gm

PRECAUTIONS:-

1. Add oxalic acid carefully so that no violent reaction takes place.
2. Mixture should be place in ice bath so that temperature does not exceed.

EXPERIMENT:-9

AIM:- Determine enthalpy of dissolution of calcium chloride solid in water at room temperature.

APPARATUS REQUIRED:- Polythene bottle, thermometer, beaker, stirrer.

CHEMICAL REQUIRED:-
Powdered calcium chloride = 8 gm

OBJECTIVE:-
The objective of the experiment is to calculate the amount of enthalpy change when substance is dissolved in large amount of solvent.

$$\text{Enthalpy of solution} = \frac{q}{w} \times mJ$$
$$Q = \text{heat change}$$
$$w = \text{weight of the substance taken}$$
$$m = \text{molecular weight}$$

PROCEDURE:-
1. Determination of water equivalent
    a. Take 100 ml of water in a polythene bottle and note the temperature after 5 – 10 minutes.
    b. Take 250 ml beaker containing water heat it to a temperature higher then room temperature.
    c. Now measure 100 ml of hot water. Measure the exact temperature of hot water then quickly add it to the polythene bottle containing 100 ml of water at room temperature. Stir and note the final temperature.
2. Determination of enthalpy of solution
    a. Take 200 ml of water in a polythene bottle for which water equivalent has already been determined. Note the temperature after 5 – 10 minutes i.e. when it becomes constant.
    b. Now add 8 gm of powdered $CaCl_2$ to the polythene bottle containing 200 ml water. Stir and note the temperature.

OBSERVATIONS:-

   (a) For water equivalent:-

Initial temperature of cold water $= t_1°C$

Temperature of hot water $\qquad = t_2°C$

Final temperature after mixing $= t_3°C$

Vol. of water taken polythene bottle $= 100$ ml $= 100$ g

Vol. of hot water added $= 100$ ml $= 100$ g

Assuming density of water $= 1$ g/ml

Applying the principle of Heat lost $=$ Heat gained

Heat lost by hot water $= 100 \times (t_2 - t_3)$ cal.

Heat gained by cold water $= (100 + w)(t_3 - t_1)$ cal.

$$100 + w = \frac{100 \times (t_2 - t_3)}{(t_3 - t_1)}$$

$$W = \left[\frac{100 \times (t_2 - t_3)}{t_3 - t_1} \right] - 100 \text{ cal.}$$

(b) Temperature of water taken $= t_1°C$

Volume of water taken $= 200$ ml $= 200$ g

Final temperature after mixing $= t_4°C$

Heat absorbed by 8.0 g of solid

(Calcium chloride) $= (200 + 8 + w)(t_1 - t_4)$

Heat absorbed by rule – 1 of solid i.e., M

$$= \frac{(200 + 8 + w)t_1 - t_4}{8} \times M \text{ Cal}$$

M = Molecular weight = a cal (say)

RESULT:-

Enthalpy of solution = a cal/mol

$= 4.184 \times$ a J/mol

PRECAUTIONS:-

1. Fresh calcium chloride should be taken otherwise it will absorb moisture from atmosphere.

2. Temperature should be noted with the help of a thermometer graduated at 0.1℃

EXPERIMENT: 10

AIM:- Determine experimentally the partition coefficient of I_2 in CCl_4 and water.

APPARATUS REQUIRED:-
Four stoppered glass bottles, burette, pipette, conical flask.

CHEMICAL REQUIRED:-
1. 2% I_2 solution in CCl_4
2. N/20, N/100, $Na_2S_2O_3$ solution
3. starch solution
4. 10% KI solution
5. Distilled water

OBJECTIVE:-
When a mixture of two immiscible liquids a solute is added, it dissolves and distributes itself in a fixed ratio in both the liquids provided the form of solute does not change during distribution.

Suppose C_1 and C_2 are the concentration of solute (I_2) in two immiscible liquids i.e., CCl_4 and H_2O. Then

Where k is partition or distribution coefficient. The values of k is different for different pairs of liquids.

PROCEDURE:-

1. Take for clean and dry glass stoppered bottles. Label them as 1,2,3,and 4. Bottles must fit tightly.
2. Take 25 ml, 20 ml, 15 ml, and 10 ml saturated solution I_2 in CCl_4 in to bottle no. 1,2,3 and 4.
3. Now fill pure CCl_4 in another burette and take 0,5,10 and 15 ml CCl_4 into Bottle no. 1,2,3 and 4 resp. So that the total volume in each bottle is 25 ml.
4. Now add 150 ml distilled water in each bottle so that the total volume in each bottle is 175 ml.
5. Stopper each bottle and shake the contents of each bottle for atleast half an hour.

6. Now keep the bottles as such till the two layers separate clearly.
7. The lower CCl_4 layer will be intense violet and the upper aqueous layer will be yellowish.
8. Separate the two layers of each bottle with the help of a separating funnet in separate beakers.
9. Now Pipette out 20ml of aqueous layer from bottle no. 1 into the conical flask containing 10 ml 10% KI solutions and 1ml starch solution. Titrate the solution against N/100 sodium thiosulphate solution ($Na_2S_2O_3.5H_2O$) using starch indicator to know the volume of titrant used. Repeat the titration with the aqueous layer of bottle no. 2,3 and 4 and record the observations.
10. Now pipette out 5ml of CCl_4 layer from bottle no. 1 into the conical flask. Add 1g solid KI, containing 10 ml of 10% KI solution and add 20ml distilled water and shake. (Don't add starch in the beginning.) Titrate it against N/20 $Na_2S_2O_3.5H_2O$ solution. Add 1ml freshly prepared starch solution when the colour of the solution in the flask is light yellow. Continue adding hypo solution till the blue colour disappear. Repeat the experiment with bottle no. 2, 3 and 4.

Bottle No.	Volume of Aqueous layer	Burette reading		Vol. of Hypo used in ml
		Initial	Final	
1.	5ml 5ml 5ml			V_1 (say)
2.	5ml 5ml 5ml			V_2 (say)
3.	5ml 5ml 5ml			V_3 (say)
4.	5ml 5ml 5ml			V_4 (say)

Bottle No.	Volume of CCl_4 layer	Burette reading		Vol. of Hypoa Used in ml
		Initial	Final	

1.	5ml 5ml 5ml			V_5 (say)
2.	5ml 5ml 5ml			V_6 (say)
3.	5ml 5ml 5ml			V_7 (say)
4.	5ml 5ml 5ml			V_8 (say)

CALCULATIONS:-

For bottle no.1

(i) Fueor aqous layer

20ml of aqueous layer $\times N_1 = V_1 \times N/100$

N_1' i.e., normality of I_2 in water layer $= V_1/20 \times 1/100$

Strength or concentration of iodine $=$ Normality $\times$ Eq. wt.

$= V_1/20 \times 1/100 \times 127 = x$ g/ litre (say)

(ii) For CCI$_4$ layer

$5 \times N_3 = V_5 \times N/20$

N_3, i.e., normality of iodine in CCI$_4$ layer $= V_5 \times 1/20 \times 1/5$

Strength or concentration of I_2 in CCI$_4$ layer $= V_5 \times 100 \times 127 = y$ g/ litre (say)

Similarly, calculate the concentration of I_2 in aqueous layer as well as in CCI$_4$ layer for bottle no. 2, 3 and 4 respectively.

Bottle No.	Conc. In H_2o layer	Conc. In CCI$_4$ layer	$K = CCCI_4/CH_2O$

1.	X g/litre	Y g/litre	$K = \frac{Y}{X}$
2.	……..............	……..............	K = ……..............
3.	……..............	……..............	K = ……..............
4.	……..............	……..............	K = ……..............

RESULT:-

The value of distribution coefficient K will come out to be nearly same in every case or we can take the average value of K.

PRECAUTIONS:-

1. Only use glass stoppers as rubber stoppers can be attacked by organic solvents.
2. Use powdered form of iodine.
3. Much care should be taken to separate the 2 layers.
4. All the bottles should be kept at a constant temperature after shaking the contents of each bottle thoroughly.

OBSERVATION:-

$$\text{Room temperature} = t^0C$$

Table:-

CALCULATIONS:-

For Bottle No.-(i)

(i)   Aquecous layer

$$\text{Vol. taken for each titration } = 5ml$$
$$\text{Normality of } I_2 \text{ in aq. Layer } = N_1$$
$$\text{Normality of } Na_2s_2O_3 = N/100$$
$$\text{Volume used (from table) } = V_1$$

Applying normality equation

$$(I_2 \text{ in aq. Layer}) \ N_1V_1 \ = \ N_2V_2(Na_2S_2O_3)$$

$$N_1{}^x5 \qquad = \ 1/100 \ {}^x \ v_1 \ = \ v_1/500$$

Strenght of I_2 = Normality ^{x} Eq. wt.

$$= v'_1/500 \ {}^x \ 127 \text{ g/l} \ = \text{ a g/litre}$$

(ii) CCI₄ layer

Volume taken for each titration = 5 ml

Normality of I_2 in CCI₄ layer	$= N_2$
Normality of $Na_2S_2O_3$	$= N/20$
Volume used	$= v''_1$

Applying normality equation

$(I_2 \text{ in CCI}_4 \text{ layer})$ $\qquad N_3V_3 \ = \ N_4V_4 \ (Na_2S_2O_3)$

$$N_3{}^x \ 5 \ = \ 1/20 \ {}^x \ v''_1$$

$$N_3 \quad = \quad v''_1/100 \ {}^x \ 127 \text{ gl} = \text{b g/l}$$

Similarly, for Bottle No. ii, iii and iv.

(i) Table:- (i): Titration of aqueous layer using N/100 $Na_2S_2\backslash O_3$

| Bottle No. | Volume Taken (ml) | Burette reading | | Vol.of $Na_2S_2O_3$ Used (ml) |
		Initial	Final	
I	5.0 5.0 5.0			

EXPERIMENT NO. 11

AIM :-
To determine the CST of phenol – water system.

APPRATUS USED :-
A boiling tube, a stirrer, a thermometer graduated to 0.1 degree, 400ml beaker, iron stand, tripod stand.

CHEMICALS REQUIRED :-
Phenol and distilled water.

OBJECTIVE :-

It is based on the fact that when water and phenol are mixed together at room temperature they form heterogeneous mixture having white turbidity. Now when the system is heated a temperature comes where turbidity disappears on cooling turbidity appears again. Noting the temperature at which turbidity disappears and at which it appears the mean of the two temperatures give the temperature of mixing of phenol water system. Now the solutions of phenol od different compositions are prepared and the temperature of miscibility of the two is determined in each case. Then a graph of composition of mixture vs. miscibility temperature is plotted. The temperature corresponding to the maximum is the CST of the system.

PROCEDURE :-
(I)Take 60gm of phenol in a previously weighed boiling tube.

(ii)Add 2ml of distilled water with the help of a graduated pipette into the phenol taken In a boiling tube. Thus the % of phenol by weight is 75%.

(iii)Fill $2/3^{rd}$ of the 400ml beaker with water and keep it on the wire gauge placed on the tripod stand.

(iv)Clamp the boiling tube into the beaker as shown. Fit the cork with two holes, one for the stirrer and other for thermometer.

(v)Heat the beaker slowly and stir the phenol water mixture. Note the temperature at which turbidity just disappears. Stop heating. Now allow thw mixture to cool and note the temperature when thw turbidity just appears.

(vi)Now again add 2ml of distilled water with the help of a graduated pipette. Thus the % of phenol by weight is 60%. Repeat step (v).

Repeat the process after adding 2ml of distilled water each time taking at least seven or eight readings.

OBSERVATIONS :-

Weight of empty boiling tube = $w_1$gm

Weight of tube + phenol = $(w_1 + 6)$gm

Weight of phenol = 6.0gm

Density of water = 1gm/ml (assuming)

RESULT :-

(i)CTS of phenol-water system = …………..^{o}C

(ii)Composition of the system

Phenol = …………..%

Water = ……………%

PRECAUTIONS :-

(i)Handle phenol very carefully as it causes severe skin burns.

(ii)Take care that in each case the level of phenol-water system in the tube must be atleast one cm below the level of water in the beaker.

(iii)For gradual and more uniform heating, surrounded the boiling tube with outer jacket (a more bigger tube).

(iv)Stirring inside the solution and outside in water must be done constantly.

(v)The bulb of the thermometer must remain dipping in phenol water system.

EXPERIMENT:- 12

AIM:- Determine solubility of benzoic acid at different temperatures and calculate ΔH of dissolution.

APPRATUS REQUIRED:-
Test tubes, Test tube holder, beaker, stand, burner, thermometer etc.

CHEMICAL REQUIRED:-
1. Benzoic acid
2. Water

OBJECTIVE:-
It was perform to check the solubility of benzoic acid. A graph is plotted between solubility an temperature and from graph solubility an any temperature can be determined by using Vant's Hoff's equation.

$$\text{Log } S_2 - \log S_1 = \text{Type equation here.}$$

PROCEDURE:-
1. Take 4 boiling test tubes and label them as 1,2,3, and 4.
2. Now weigh 0.1 gm, 0.15 gm, 0.20 gm and 0.25 gm of benzoic acid and transfer to the labeled tube.
3. Add 20 ml of water to each tube and place them in beaker containing water.
4. Now stir the contents of tube 1 and note the temperature at which benzoic acid dissolves completely.
5. Similarly note down the temperature of all tubes.

OBSERVATIONS:-

Test tube No	Temperature at Which benzoic acid dissolves	Amount Taken(g)	Solubility Mole/litre
I	t_1	0.10	$\frac{0.1}{122} \times 50$
II	t_2	0.15	$\frac{0.15}{122} \times 50$

III	t_3	0.20	$\frac{0.20}{122} \times 50$
IV	t_4	0.25	$\frac{0.25}{122} \times 50$

CALCULATION:-

Plot a graph between solubility and temperature from the graph determine the solubility of benzoic acid using vant's Hoff's equation.

PRECAUTIONS:-

1. Handle the acid carefully.
2. Temperature should not exceed.

EXPERIMENT:-13

AIM :-
To determine the enthalpy of neutralization of a weak acid (say acetic acid) versus strong base (say NaOH) and determine the enthalpy of ionization of the weak acid.

APPRATUS USED :-
Polyethene bottles thermometer, stirrer *, beakers etc*

CHEMICALS REQUIRED :-
$\frac{N}{2}$ acetic acid, $\frac{N}{2}$ $NaOH$, distilled water.

OBJECTIVE :-
It is used to determine the value of enthalpy of neutralization which is defined as enthalpy change accompanying when one gram equivalent of the acid (or base) by base (or an acid) in a dilute solution for strong acids and for strong bases its value comes out to be nearly -58 kJ. It is nearly same for both strong acids and strong bases because in this case all acids and bases are almost completely ionised.

PROCEDURE :-
Determination of water equivalent of polyethene bottle.

(i)Take 100ml of distilled water in polyethene bottle fitted with a stirrer and a thermometer. The thermometer should be 1cm above the bottom of the bottle. When thermal equilibrium is achieved then note down the temperature say it is t_1^oC.

(ii)Heat 150ml of distilled water in a 250ml beaker at a temperature which is about 10-20°C higher than the room temperature.

(iii)Take 100ml of hot water, note down its temperature (say it is t_2^oC) and immediately transfer it into the polyethene bottle already containing 100ml of cold water. Stir well and note down the temperature of water(after mixing hot and cold water) say it is t_3^oC.

(iv)Throw away water from the polyethene bottle and cool the room temperature.

(v)Take 100ml of 0.5 N acetic acid in a polyethene bottle fitted with a rubber cork with two holes. Insert a thermometer into one hole and a stirrer into the second hole.

(vi)Place 100ml of 0.5 N NaOH in another similar polyethene bottle.

(vii)The temperature of each solution is noted separately. Let these are t_4^oC and t_5^oC respectively.

(viii)transfer 100ml 0.5 N NaOH into the acid as quickly as possible. The mixture is well stirred.

(ix)The temperature is noted after every minute till a constant temperature is attained. Let it be t_3^oC.

OBSERVATIONS :-
(i) Determination of water equivalent

$$100 \ (t_2 - t_3) = (100 + w) \ (t_3 - t_1)$$

$$\text{Water equivalent, w} = \left[\frac{100(t2 - t3)}{(t3 - t1)} \right] - 100$$

(ii)Determination of enthalpy of neutralization

$$\text{Initial temperature of acid} = t_4^OC$$

$$\text{Initial temperature of base} = t_5^oC$$

$$\text{Final temperature after neutralization} = \left(t6 - \frac{t4 + t5}{2} \right) - 100$$

Water equivalent of polyethene bottle = w g

PRECAUTIONS :-
(I)Temperature should be noted using thermometer graduated to 0.1^oC.

(II)The mixture of acid and base should be well stirred.

EXPERIMENT:-14

AIM :- Detection of organic compounds.

I. PRELIMINARY TESTS FOR ORGANIC COMPOUNDS

PHYSICAL CHARACTERSTICS

(a) Colour – from the colour of organic compounds following observation can be drawn

Experiment	Observation	Inference
(a) Note the colour of compound	I. Yellowish II. Pale yellow liquid III. Deep orange yellow	Nitro compounds Nitrobenzene Nitro aniline, nitrophenols.

(b) Odour:- From the odour of compound following observation can be drawn

Experiment	Observation	Inference
(a) Note the odour of the compound	(a) Fruity smell (b) Vinegar like smell (c) Pungent smell (d) Wine like smell (e) Fishy smell	Esters Acetic acid Benzoyl chloride Alcohol Amines

(c) Ignition Test:- On burning organic compounds give following information

Experiment	Observation	Inference
(a) Take a small amount of compound on a nickel spatula and heat.	a) Burns with a smoky flame. b) Burns with non smoky flame. c) Burnt with sugar smell. d) Ammonical smell. e) Irritating smell with coughing. f) rotten eggs smell	Aromatic compounds Non aromatic compounds Carbohydrates Urea Benzoic acid, succinic acid, salicyclic acid Sulphur present

(d) Solubility Test:- The organic compound either dissolved in water or NaOH   or dil. HCl gives following information

Experiment	Observation	Inference
a) Take a small amount of compound in a clean test tube and add 3 – 4 ml water. Shake well.	a) Sparingly soluble or insoluble b) Soluble	Hydrocarbons, esters, ethers etc. Lower alcohols, aldehydes, ketones, acids etc.
b) Test the compound with litmus.	c) Turns blue litmus to red	Acids, phenols
	d) Turns red to blue	Lower amines
c) Test the solubility in dil. HCl	e) Soluble and reprecipitated on adding alkali	Base
d) Test the solubility in NaOH	f) Soluble but reprecipitated on adding acid	Phenol

PRELIMINARY CHEMICAL TESTS

1. **Sodalime test:-** On mixing organic compound with sodalime following observation are seen

Experiment	Observation	Inference
a) Take about 0.5 gm of solid compound with 2 gm of sodalime (NaOH + CaO) in a test tube and heat it	a) Ammonical smell b) Burnt sugar smell c) Smell of phenol d) Smell of bitter almonds	Urea, acetamide Carbohydrates Phenolic acid Benzaldehyde derivatives

2. **Ferric chloride test:-** with ferric chloride following observation are seen

Experiment	Observation	Inference
a) Dissolve 0.5 gm of compound in 2 – 3 ml of water and add 4- 5 drops of neutral FeCl₃	(a) Violet (b) Blue (c) Blue violet	Phenols, salicyclic acid P-cresol Resorcinol, m-

	(d) White ppt. changing to violet	cresol α-napthol
solution		

3. Conc. H_2SO_4 test:-

Experiment	Observation	Inference
a) Heat 0.5 gm of compound with 2 – 3 ml of conc. H_2SO_4	a) Dissolves with violent reaction.	Alcohols
	b) Soluble in cold water.	Ethers
	c) Dissolves without charring and CO is given out.	Formic acid or oxalic acid
	d) Charring with no gas is evolved.	Aldehydes, ketones and aromatic hydroxy acid Urea, oxalate
	e) Dissolves without charring and CO_2 is given out.	Aromatic
	f) Dissolves slowly but not precipitated on dilution.	Carbohydrates
	g) Dissolves on heating with charring CO_2	

4. **Sodium bicarbonate test:-**

Experiment	Observation	Inference
a) Mix the compound in water and add solid $NaHCO_3$	a) Evolution of CO_2 with effervescence.	Acid
	b) Dissolves with no evolution of CO_2 gas	Phenols

5. **Action of $KMnO_4$ solution:-**

Experiment	Observation	Inference
a) Add a very dil.	a) Decolorised	Unsaturated

Solution of $KMnO_4$ and dil. H_2SO_4 to the solution of compound	immediately b) Decolorised slowly	compounds Aldehydes

DETECTION OF EXTRA ELEMENTS

1. **For detection of extra elements we have to prepare Lassaigne's extract**
   a. **Preparation of lassaigne's extract:-**

   Cut small pieces of dry sodium metal into ignition test tube now heat it till the pieces changes to silver globule. Now add a pinch of organic compound into this test tube again heat it till the tube becomes red hot. Now take a china dish containing 10 ml water now break the hot tube into this china dish. Now the solution is heated and filtered. The filtrate is known as lassaign's extract. (L.E.) or sodium extract (S.E.)

Experiment	Observation	Inference
a) Test for nitrogen:- To about 2ml of the S.E., added a few drops of NaOH solution, followed by the addition of about 2 ml $FeSO_4$ solution. Boiled the solution and add dill. H_2SO_4 and shake.	No deep blue or blush green colour	Nitrogen absent
b) Test for Sulphur:- Add two drops of sodium nitroprusside solution to about 1ml of Lassaigne's extract.	No violet colour	Sulphur absent

c) Test for Halogens:-
  I. Belistein's Test:-
     Heated a copper wire in the non-lumionous flame, till it imparts no colour. Dipped the copper wire into the organic compound and heated again in the flame.
	A green colour	Halogens present

II. AgNO₃ Test:- Added a fw drops of conc.	a) No white ppt.	Cl absent

HNO_3 to about 5ml of Lassaigne's extract. Boiled off all gases. The solution is cooled and treated with $AgNO_3$ solution.	b) No light yellow ppt. c) Yellow ppt. insoluble in NH_4OH.	Br absent I confirmed

IDENTEIFICATION OF FUNCTIONAL GROUP

1. Test for – COOH group:-

Experiment	Observation	Inference
a) Sodium bicarbonate test:- to the given compound add a saturated solution of sodium bicarbonate	a) Dissolve with brisk evolution of CO_2 gas.	-COOH group confirmed
b) Ester test:- To a small amount of organic compound add 1 – 2 ml alcohol and 2 -3 drops of conc. H_2SO_4	b) Fruity smell	-COOH group confirmed

2. Test for phenolic group:-

Experiment	Observation	Inference
a) Litmus test:- add few drops of blue litmus solution to aqueous compound	a) Blue litmus turned red	-COOH or Phenolic group present
b) Ceric ammonium nitrate test:- To aqueous solution of organic compound add few drops of ceric ammonium nitrate.	b) Brown ppt.	Phenolic group confirmed

3. Test for Ketonic group $> C = O$:-

Experiment	Observation	Inference
a) Sodium nitroprusside	a) Presence of red	Presence of

	colour	$> C = O$ group
test:- add a pinch of organic compound in alkaline sodium nitroprusside solution. b) Schiff's reagent test:- add small amount of organic compound to $1 - 2$ ml of schiffs reagent and shake.	b) Pink colour	Presence of $> C = O$ group Or Aldehyde present

4. Test for Carbohydrates:-

Experiment	Observation	Inference
a) Molisch test:- add few drops of alcoholic α-naphthol aolution to about 1 ml of aqueous solution of organic compound b) Conc. H_2SO_4 test:- add about 1 ml of con. H_2SO_4 to the given compound and warm.	a) Deep violet ring at the junction b) Charring	Carbohydrates present Carbohydrates present

5. Test for esters:-

Experiment	Observation	Inference
a) Hydrolysis Test:- To about 1 ml of substance, a drop of phenol phthalein and few drops of dil NaOH are added. Heat the contents on a water bath.	a) Pink colour disappears	Ester group present

6. Test for amide (-CONH$_2$) group:-

Experiment	Observation	Inference
a) HNO$_2$ test:- add a pinch of organic	a) Effervescence	-CONH$_2$ group present

Experiment	Observation	Inference
compound to ice cold solution of $NaNO_2$ and dil. CH_3COOH b) NaOH test:- Heat a small amount of compound with NaOH solution	b) NH_3 (g) Evolved	$-CONH_2$ group present

7. Test for – NH_2 group:-

Experiment	Observation	Inference
a) HNO_2 test:- To ice cold solution of compound in dil. HCl. Add few drops of saturated $NaNO_2$ solution.	a) Brisk effervescence	Aliphatic $-NH_2$ group present
b) Dye test:- To about 0.5 g of compound add 2-3 ml of water containing 1 ml of conc. HCl. Then cool the contents in ice, add 2 ml of 10% $NaNO_2$ solution dropwise with constant stirring then add ice cold solution of alkaline β-naphthol	b) Orange-red dye	Aromatic D($-NH_2$) group present

8. Test for anilides:-

Experiment	Observation	Inference
a) NaOH Test:- Heat a small amount of compound with 1-2 ml concentrated solution of NaOH	a) Peculiar smell	-NHCOR (anilide) group present

9. Test for –No_2 group:-

Experiment	Observation	Inference

Experiment	Observation	Inference
a) Dye Test:- Heat the given compound with 1 ml of conc. HCl and few pieces of granulated tin and heat the mixture for about 5 minutes in a boiling water bath. Filters and cool the filterate in ice bath, then add 1, 2 – ml of $NaNO_2$ solution followed by 1, 2 – ml of ice cold alkaline β-naphthol solution.	a) Orange red	-NO_2 group

10. Test for halogen group:-

Experiment	Observation	Inference
a) Boil about 0.2 or 2-3 ml of compound with 2-3 ml of KOH or NaOh (alc.) for 5 minutes. Cool and add dil. HNO_3 and $AgNO_3$.	a) Ppts. Formed	Halogen group is present

11. Test for –OH (alcoholic) group:-

Experiment	Observation	Inference
a) Ester test:- add 1g of CH_3COONa to about 1-2 ml of compound and then 2-3 drops of conc. H_2SO_4	a) Fruity smell	Alcoholic group present
b) Ceric ammonium nitrate test:- To few drops (10-15), add 2 ml of ceric ammonium nitrate	b) Red or pink colour	Alcoholic group confirmed

solution		

12. Test for – CHO group:-

Experiment	Observation	Inference
a) Fehiling's solution test:- add a small amount of organic compound to 2-3 ml of fehling's solution. Heat the contents on a water bath.	a) Red ppts.	CHO group present
b) Tollen's reagent test:- Warm 4-5 ml of Tollen's reagent with small amount of organic compound on a water bath.	b) Silver mirror is formed	-CHO group present
c) Sodium bisulphite test:- To about 1 ml of organic compound, add 1-2 ml of saturated sodium bisulphite solution.	c) White ppts.	-CHO group $> C = O$ group present

PREPARATION OF SOLID DERIVATIVES

1. **DERIVATIVES OF PHENOLS:-**
   a. Picrates: - Mix equal amounts of saturated solution of compound and picric acid in benzene (say 3 ml each). Shake the contents vigorously. Filter the precipitates formed and recrystallise from benzene (if required).

2. **DERIVATIVES OF CARBOXYLIC ACIDS:-**
   a. S-Benzylisothiruronium salts :- Dissolve 0.5 g of given compound in 5-6 ml of water by heating. Add a drop of phenolphthalein indicator and then add NaOH till it becomes pink. Add 1-2 drops of HCl. Dissolve approximately 2.0 g of S-Benzyl isothiuronium chloride in 5-6 ml of water. Mix both the solution stir and cool the precipitates formed are filtered and crystallized from hot water.

3. **DERIVATIVES OF CARBOHYDRATES:-**
   a. Osazones :- Shake about 1 g of compound, 2 g of phenyl hydrazine hydrochloride and 3g of sodium in a boiling water bath.
   Yellow mass separates out, recrystallise from alcohol.
   Glucose, fructose and sucrose form osazone in approximately 2, 5 and 30 minutes respectively.

4. **DERIVATIVES OF ALDEHYDES AND KETONES:-**
   a. 2 ,4- Dinitrophenyl hydrazone: - Add approximately 5 ml of 2, 4-Dinitro hydrazine to 1g or 1ml of the compound in a dry test tube. Then add 1-2 drops of conc. H_2SO_4 Shake the contents and heat for few minutes. Cool, filter and recrystallise from alcohol or benzene.