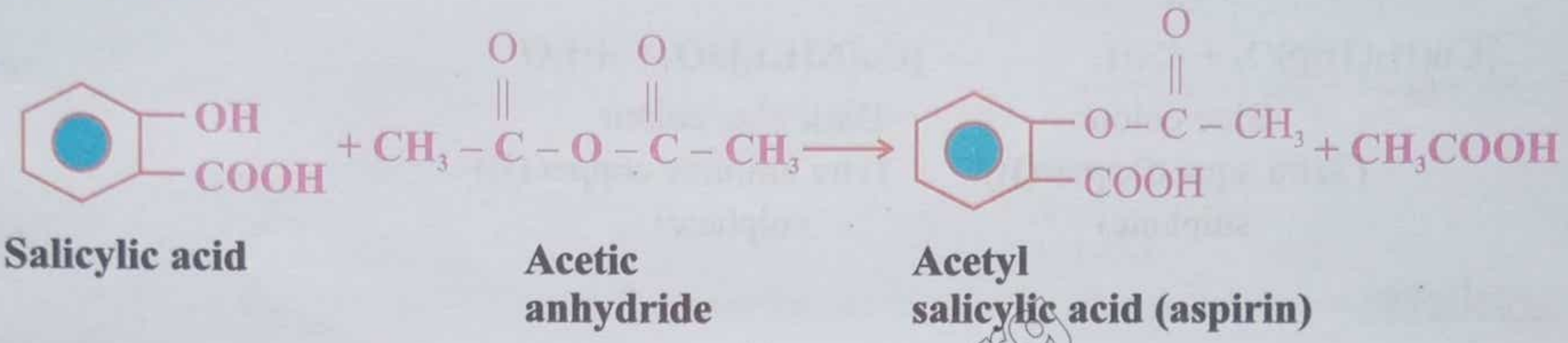
EXPERIMENT No-1

To prepare a pure sample of aspirin.

Material and Apparatus Required:

Salicylic acid (10g), acetic anhydride (10 cm^3), acetic acid (10 cm^3), ice cold water, round bottom flask, reflux condenser, tripod stand, wire gauze and burner.

Reaction:



Procedure:

Take 10g of salicylic acid in a round bottom flask. Add 10 cm^3 of acetic acid and 10 cm^3 of acetic anhydride in it. Boil this mixture under reflux for 30 minutes. Stop heating and add about 200 cm^3 of ice cold water in it with vigorous stirring. White crystals of aspirin will be formed. Filter the crystals, dry them and show to the examiner.

The melting point of acetyl salicyclic acid (aspirin) is 136—137 °C.

VIVA VOCE

1. 10 cm^3 acetic acid and 10 cm^3 acetic anhydride used in aspirin preparation is called as **acetylating mixture.**
2. Aspirin is an **aromatic substance.**
3. The chemical formula of aspirin is **HOOC C_6H_4 $OCOCH_3$.**
4. Aspirin is used as:
   - *(a)* **Analgesic** (pain reliever)
   - *(b)* Anti-inflammatory and initial drug of choice for treatment of arthritis.

EXPERIMENT No-2

To prepare a pure sample of copper-ammine complex.

Apparatus and Chemical Required:

$CuSO_4$ solution, NH_4OH, ethanol, 100 cm^3 beaker, pipette.

Reaction:

$$[Cu(H_2O)_4]SO_4 + 4NH_3 \longrightarrow [Cu(NH_3)_4]SO_4 + 4H_2O$$

Blue colour (Tetra aqua copper (II) sulphate)	Dark blue colour Tetra ammine copper (II) sulphate)

Procedure:

Take 10 cm^3 of saturated solution of $CuSO_4$ in 100 cm^3 beaker. Add ammonium hydroxide solution in it, drop wise. Firstly green colour appears, add more NH_3 till a dark blue colouration is obtained. Now add ethanol in it, till dark blue crystals of tetra ammine copper (II) sulphate $[Cu(NH_3)_4]SO_4$ are formed. Filter the crystals, dry them and then show to the examiner.

VIVA VOCE

1. In copper ammine complex NH_3 is a **ligand** which donate electron pair to Cu^{+2}.
2. In cooper ammine complex (tetra ammine copper(II) sulphate) **coodrination number is four**.
3. The **oxidation state of copper is +2** in copper ammine complex.
4. Copper ammine complex is used in **dyeing and printing purpose**.
5. Copper ammine complex has dsp^2 hydridization and its structure is **square planar**

EXPERIMENT No-3

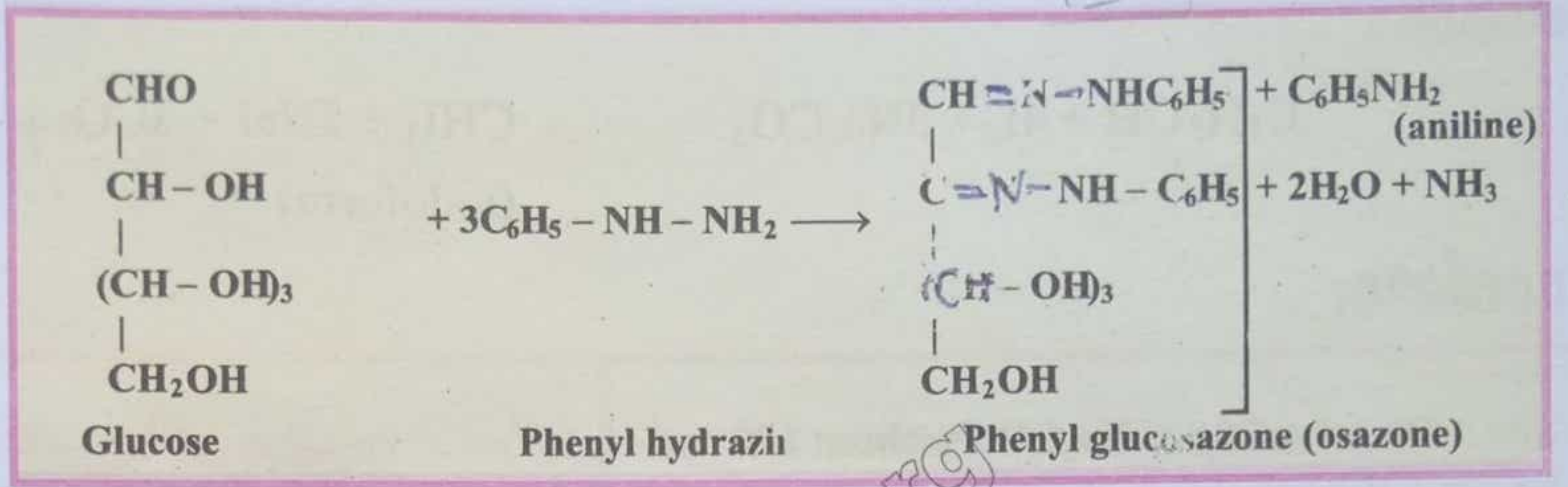
To prepare a pure sample of glucosazone.

Apparatus and Chemical Required:

Round bottom flask, tripod stand, burner, wire guaze, water bath, phenyl hydrazine hydrochloride (3g), sodium acetate (3g), glucose (1g).

Reaction:

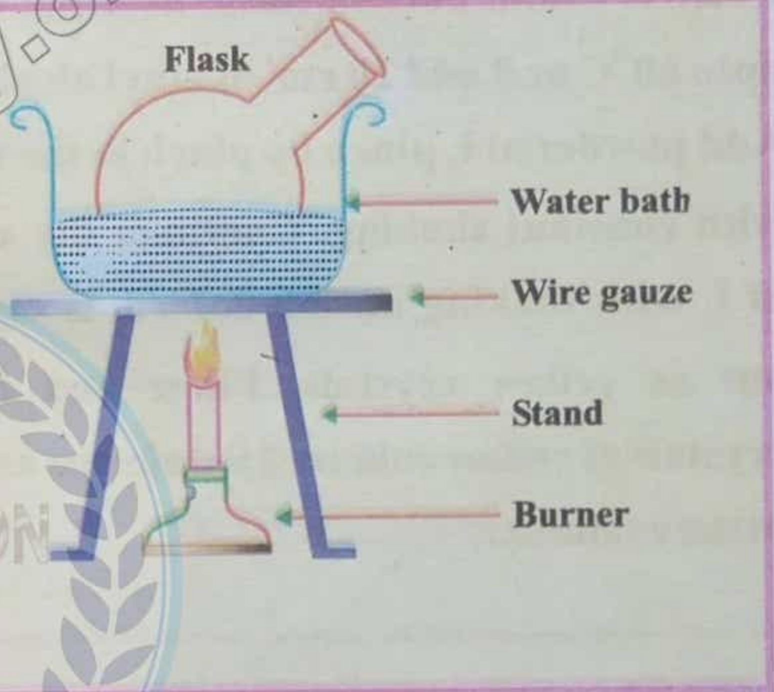
(Imp)



$$\begin{array}{l} CHO \\ | \\ CH-OH \\ | \\ (CH-OH)_3 \\ | \\ CH_2OH \end{array} + 3C_6H_5-NH-NH_2 \longrightarrow \left.\begin{array}{l} CH=N-NHC_6H_5 \\ | \\ C=N-NH-C_6H_5 \\ | \\ (CH-OH)_3 \\ | \\ CH_2OH \end{array}\right] \begin{array}{l} + C_6H_5NH_2 \\ \quad\text{(aniline)} \\ + 2H_2O + NH_3 \end{array}$$

Procedure:

Dissolve 3g of phenyl hydrazine hydro-chloride in 10 cm^3 of water containing 3 grams of sodium acetate in a clean round bottom flask. Warm gently on a water bath and shake the contents of flask until it dissolved. Make a solution of 1g of glucose in 5 cm^3 water in a test tube separately. Add glucose solution into the round bottom flask and stir the contents of flask constantly. Place the flask in boiling water bath. After about 20 minutes, yellow crystals of phenyl glucasozone begins to separate. Cool down the contents of the flask and separate the yellow crystals of phenyl glucosazone by filteration.



Dry the crystals and show to the examiner.

VIVA VOCE

1. Glucose is a **polyhydroxy aldehyde**.
2. Glucose is a **reducing sugar**.
3. Phenyl hydrazone test is used to **identify the sugars**.
4. Preparation of glucasozone involves **nucleophilic substitution reaction**.
5. Phenyl hydrazine is a **reducing agent**.

EXPERIMENT No-5

Determine the concentration of Barium ions (Ba^{2+}) per dm^3 in the given solution.

Theory:

Ba^{2+} ions can be estimated as $BaCrO_4$ by adding K_2CrO_4 solution to an ammonical solution of a soluble salt of Barium.

Reaction:

$$BaCl_2 + K_2CrO_4 \longrightarrow \underset{\text{(Yellow ppts)}}{BaCrO_4} + 2KCl$$

Procedure:

Take 10 cm^3 of the given solution of Ba^{2+} ions in a 250 cm^3 beaker. Dilute the solution to 50 cm^3 with distilled water and then add NH_4OH till it smells ammonia. Then add 10% solution of K_2CrO_4 till the supernatent layer stops giving any yellow ppt. on addition of K_2CrO_4 solution. Stir the solution well and allowed to stand for 15 minutes. Filter the precipitate in a pre-weighed filter paper. Dry the precipitate in electric oven at 115 c° for 30 minutes and calculate the mass of $BaCrO_4$.

Supposed Calculations:

Volume of sample taken = 10 cm^3

Mass of filter paper = 1.0 g (supposed)

Mass of filter paper + $BaCrO_4$ = 1.5 g (supposed)

Mass of $BaCrO_4$ = 1.5 − 1.0 = 0.5 g

253.33 g of $BaCrO_4$ contains Ba^{2+} = 137.33 g

0.5 g of $BaCrO_4$ contains Ba^{2+} $= \frac{137.3}{253.3} \times 0.5$

$= 0.271$ g

10 cm^3 of given solution contains Ba^{2+} = 0.271 g

1000 cm^3 of the solution will contains Ba^{2+} $= \frac{0.271}{10} \times 1000$

$= 27.1$ g

Result:

The given solution contains 27.1 g Ba^{2+} ions per dm^3 of the solution.

(253.33 g is the formula mass of $BaCrO_4$ and 137.33 g is the atomic mass of Ba)

EXPERIMENT No-4

To prepare a pure sample of iodoform.

Apparatus and Chemicals Required:

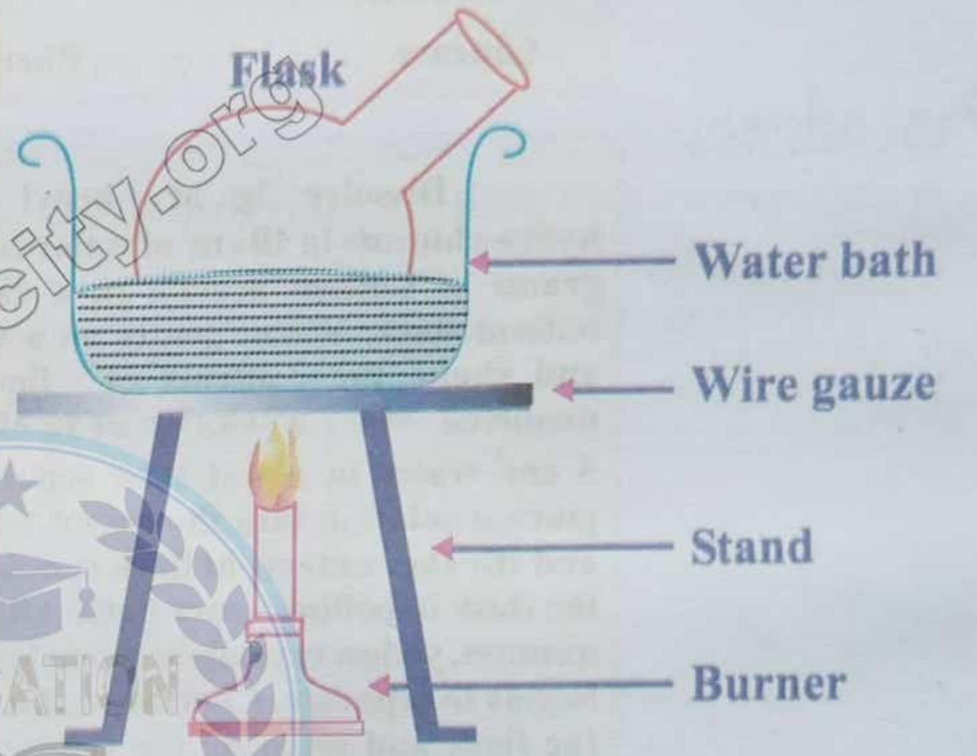
Round bottom flask, water bath, wire guaze, tripod stand, burner, ethyl alcohol (20 cm^3), sodium carbonate (20g) and iodine (10g).

Reaction:

$$C_2H_5OH + 4I_2 + 3Na_2CO_3 \longrightarrow \underset{\text{(iodoform)}}{CHI_3} + 5NaI + 3CO_2 + 2H_2O + HCOONa$$

Procedure:

Dissolve 20g of Na_2CO_3 in about 100 cm^3 water is round bottom flask, heat the solution upto 60°C and add 20 cm^3 of ethyl alcohol in it. Add powdered I_2 pinch by pinch in the mixture with constant shaking. Continue the addition of I_2 with shaking until iodoform is separated out as yellow crystals. Filter and dry the crystals of yellow coloured iodoforms and show to the examiner.



VIVA VOCE

1. Iodoform (CHI_3) is an example of haloforms other include chloroform $CHCl_3$ and bromoform $CHBr_3$.
2. Iodoform (CHI_3)can also be prepared from acetone, acetaldehyde or any other methyl ketone.
3. Iodoform is of yellow colour.
4. Iodoform is used in medicine like iodine tincture, iodex, etc.

EXPERIMENT No-6

To detect the elements (N, S and halogens) present in an organic compound.

Principle:

Organic compounds having N, S and halogen when strongly heated with sodium metal, converted into ionic compounds. e.g.,

For N-element:

$$Na + C + N \longrightarrow NaCN \quad \text{(Sodium cyanide)}$$

For S-element:

$$2Na + S \longrightarrow Na_2S \quad \text{(Sodium sulphide)}$$

For halogens:

$$2Na + X_2 \longrightarrow 2NaX \quad \text{(Sodium halide)}$$

Preparation of Sodium Extract (Lassaigne's Solution):

Take a small piece of Na metal in a fusion tube and heat upto melting of sodium. Add small amount of organic compound in fusion tube. Heat it till red-hot. Plunge fusion tube into china dish containing about 20 cm^3 of distilled water. Stir the solution and filter it. The filterate is called sodium fusion filterate or Lassaigne's filterate or sodium extract. Divide it into three parts.

Experiments	Observations	Inferences
Test for Nitrogen:	(Case-I)	
2 cm^3 of Na -extract + few drops of NaOH solution + fresh $FeSO_4$ solution + Acidified $FeCl_3$ solution + dil. H_2SO_4	Prussian blue or green colouration or ppt. is formed	Nitrogen is indicated
	(Case-II) Blood red coluration	Nitrogen and sulphur both are indicated.

Experiments	Observations	Inferences
Test for Sulphur:		
1. 2 cm^3 of Na-extract + acetic acid (CH_3COOH) + lead acetate ($Pb(CH_3COO)_2$)	Black ppt. formed	Sulphur is indicated.
2. 2 cm^3 of Na -extract + few drops of sodium nitroprusside $Na_2[Fe(CN)_5NO]$	Violet colouration	Sulphur is present.
Test for Halogens:		
2 cm^3 of sodium extract + 2 ml conc. HNO_3 + boil + cool + $AgNO_3$ solution	i. White ppt. soluble in NH_4OH	Cl is present.
	ii. Pale yellow ppt. partially soluble in NH_4OH	Br is present.
	iii. Yellow ppt. insoluble in NH_4OH	I is present.

CHEMICAL REACTIONS:

Test for Nitrogen:

Case-I:

$$6NaCN + FeSO_4 \longrightarrow Na_4[Fe(CN)_6] + Na_2SO_4$$

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

Ferric-ferrocyanide
(Prussian blue colour)

Case-II:

$$6NaSCN + FeSO_4 \longrightarrow Na_4[Fe(SCN)_6] + Na_2SO_4$$

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

Ferric-ferrothiocyanide
(Blood red colouration)

Test for Sulphur:

(i) $$Na_2S + Pb(SH_3COO)_2 \longrightarrow PbS \downarrow + 2CH_3COONa$$

(Black ppt.)

(ii) $$Na_2S + Na_2[Fe(CN)_5NO] \longrightarrow Na_4[Fe(CN)_5NOS]$$

(Violet colouration)

Test for Halogens:

(i) $NaCl + AgNO_3 \longrightarrow AgCl\downarrow + NaNO_3$
(White ppt.)

(ii) $NaBr + AgNO_3 \longrightarrow AgBr\downarrow + NaNO_3$
(Pale yellow ppt.)

(iii) $NaI + AgNO_3 \longrightarrow AgI\downarrow + NaNO_3$
(Yellow ppt.)

VIVA VOCE

1. Sodium metal reacts with air (oxygen) and moisture at room temperature, so it is **stored in Kerosene oil**

2. Sodium fusion filterat **alkaline** due to the formation of NaOH as

$Na + O_2 \longrightarrow Na_2O$ (during combustion)

$Na_2O + H_2O \longrightarrow 2NaOH$ (in china dish)

3. For the detection of halogens with $AgNO_3$ **conc. HNO_3 is added** to remove cyanide (CN^-) and sulphide (S^{2-}) if present in the solution e.g.,

$Na_2S + 2HNO_3 \longrightarrow 2NaNO_3 + H_2S\uparrow$

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

Both CN^- and S^{2-} interfere with $AgNO_3$ and form white ppt. of AgCN and black ppt. of Ag_2S respectively

$Na_2S + 2AgNO_3 \longrightarrow 2NaNO_3 + Ag_2S\downarrow$ (black)

$NaCN + AgNO_3 \longrightarrow NaNO_3 + AgCN\downarrow$ (white)

EXPERIMENT No-7

To detect the functional group present in the given organic compound (aldehyde, phenol and carboxylic acid)

Theory:

The compound containing of aldehyde as functional group are neutral. Phenol and carboxylic acid are acidic in nature.

Preliminary Identification Tests for Functional Groups:

Dissolve some amount of compound in the water and check it with litmus paper.

Test for Halogens:

(i) $NaCl + AgNO_3 \longrightarrow AgCl\downarrow + NaNO_3$ (White ppt.)

(ii) $NaBr + AgNO_3 \longrightarrow AgBr\downarrow + NaNO_3$ (Pale yellow ppt.)

(iii) $NaI + AgNO_3 \longrightarrow AgI\downarrow + NaNO_3$ (Yellow ppt.)

VIVA VOCE

1. Sodium metal reacts with air (oxygen) and moisture at room temperature, so it is **stored in Kerosene oil**

2. Sodium fusion filterat **alkaline** due to the formation of NaOH as

$Na + O_2 \longrightarrow Na_2O$ (during combustion)

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$Na_2S + 2HNO_3 \longrightarrow 2NaNO_3 + H_2S\uparrow$

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

Both CN^- and S^{2-} interfere with $AgNO_3$ and form white ppt. of AgCN and black ppt. of Ag_2S respectively

$Na_2S + 2AgNO_3 \longrightarrow 2NaNO_3 + Ag_2S\downarrow$ (black)

$NaCN + AgNO_3 \longrightarrow NaNO_3 + AgCN\downarrow$ (white)

EXPERIMENT No-7

To detect the functional group present in the given organic compound (aldehyde, phenol and carboxylic acid)

Theory:

The compound containing of aldehyde as functional group are neutral. Phenol and carboxylic acid are acidic in nature.

Preliminary Identification Tests for Functional Groups:

Dissolve some amount of compound in the water and check it with litmus paper.

Test for Aldehydes, Phenol and Carboxylic Acid

Test for Aldehydes

Experiment	Observation	Inference
1. Litmus Test: Dissolve the organic compound in water and check it with Litmus Solution.	Timus solution remains unchanged	May be aldehyde
4. Silver Mirror Test: Take 5 ml of $AgNO_3$ add very dilute NH_4OH till ppt. formed dissolves add organic compound and boil on water bath.	A silver mirror is formed	Aldehydes are confirmed.
5. Fehlings Solution Test: Add Fehling's solution to the solution of organic compound and warm.	A red ppt. of Cu_2O is formed.	Aldehydes confirmed.
6. Benedict's Solution: Take 0.2g of organic compound in test tube and add about 10 cm^3 of benedict solution in it and heat it.	Red ppt. is formed (Cu_2O)	Aldehydes confirmed.

Test for Phenol

Experiment	Observation	Inference
1. Litmus Test		
Dissolve organic compound in water and check it with litmus paper.	Blue litmus paper turns red.	May be phenol or carboxylic acid.
2. Effervescence Test:		
Take 5 cm^3 of $NaHCO_3$ solution in a test tube and add one pinch of organic compound in it.	No effervescence	Carboxylic acid absent and phenol may be present

3. Neutral $FeCl_3$ Test:		
Add neutral $FeCl_3$ solution to organic compound solution.	Violet / blue or green colour is formed.	Phenolic group is confirmed.
4. Bromine Water Test:		
Treated the organic compound with bromine water.	Colour discharges but on heating white ppt. formed.	Phenolic group is confirmed.

TEST FOR CARBOXYLIC ACID:

Experiments	Observations	Inferences
1. Litmus Test		
Dissolve the organic compound in water and check it with Litmus Solution.	Blue titmus solution turns red.	May be phenol or carboxylic acid is present.
2. Effervescence Test:		
Take 5 cm^3 of $NaHCO_3$ solution in a test tube and add one pinch of organic compound in it.	CO_2 gas evolved with effervescence.	Carboxylic acid is indicated.
3. Ester Test (Esterification)		
Take some organic compound in a test tube. Add C_2H_5OH and conc. H_2SO_4 in it and heat gently.	A fruity smell due to the formation of an ester.	Carboxylic acid is confirmed.
4. Neutral $FeCl_3$ Test:		
Add neutral $FeCl_3$ solution to the solution of organic compound.	A red or yellow ppt. is formed.	Carboxylic acid is confirmed.

VIVA VOCE

1. A mixture of $CuSO_4$, sodium carbonate and sodium citrate is called **benedict solution**.
2. A mixture of $CuSO_4$, sodium carbonate and sodium tartrate is called F**heling's solution**.
3. Aldehydes behaves as **reducing agents** in tollen test or in benedict solution test.
4. An atom or group of atoms which confers characteristic properties to organic compound is called **functional group**.
5. **Phenols** give **violet, blue or green colour** with neutral $FeCl_3$ solution whereas **carboxylic acid** gives **red or yellow ppt**.

$$\text{Heat of neutralization} = \frac{(m_1s_1 + m_2s_2)\,\theta}{1000} \times \frac{1000}{V \times M}$$

Where m_1 = mass of calorimeter
s_1 = specific heat of calorimeter
m_2 = mass of solution
s_2 = specific heat of dilute solution
θ = Rise in temperature
V = volume of acid or base used
M = Molarity of acid or base used

Experiment:

Determine the Heat of Neutralization of a Strong Acid with Strong Base:

Apparatus: *Calorimeter, Beakers, thermometer, weight box, balance stopwatch measuring cylinder.*

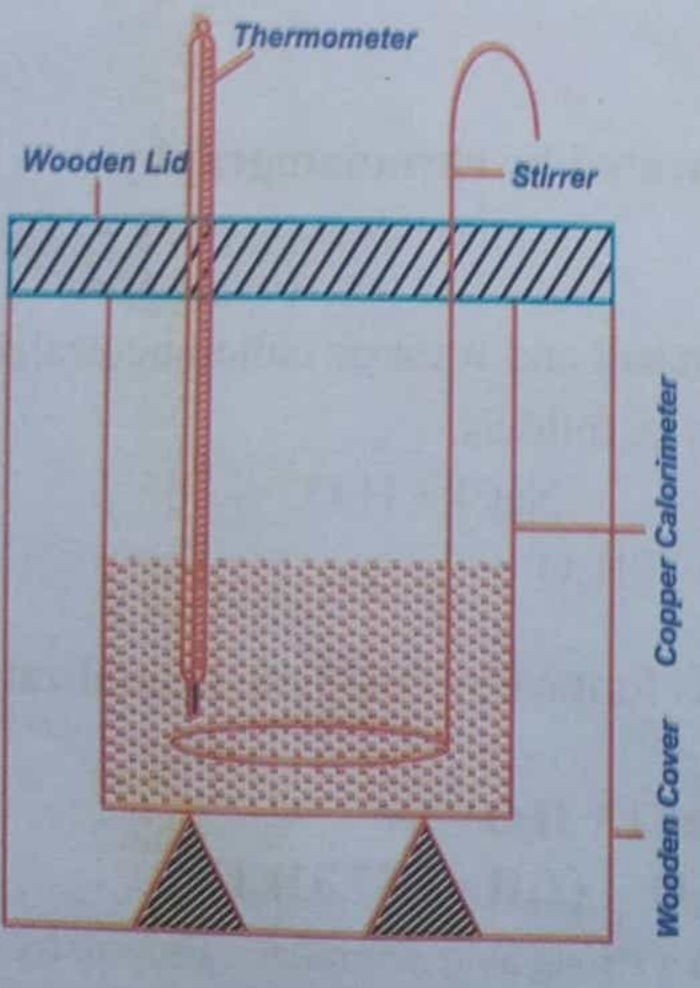
Chemical: **1M HCl, 1M NaOH**

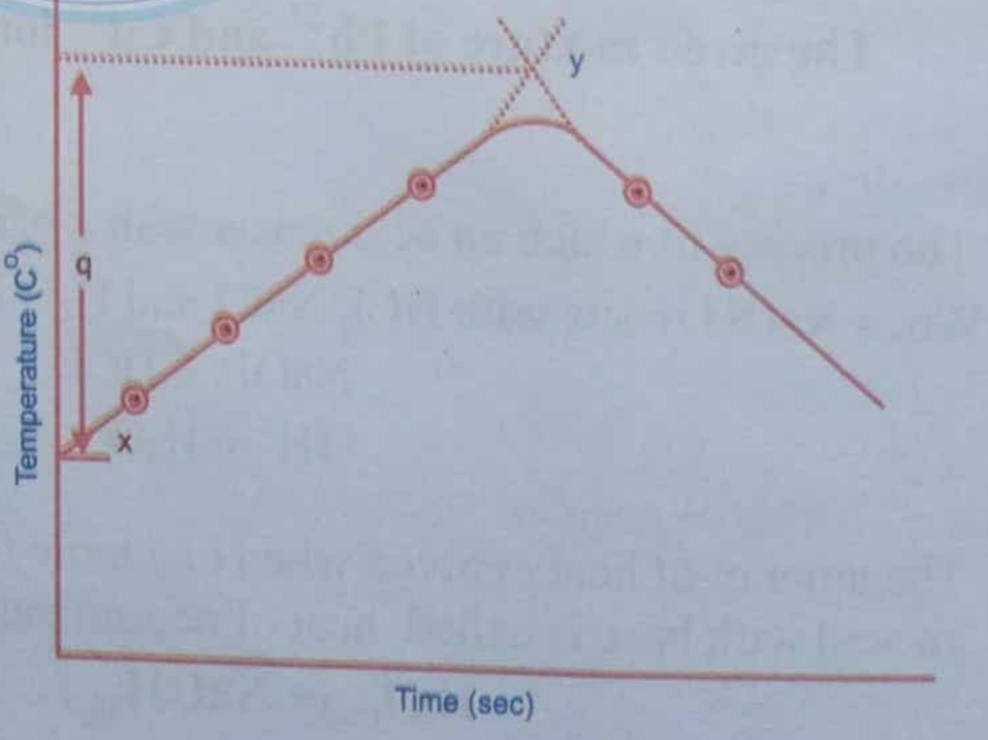
Procedure:

For preparing approximately IM NaOH, take 4 gm of it, and dissolve it in 100 cm^3 of solution and for preparing approximately IM HCI, take 10 cm^3 of conc. HCI and dilute it to 100 cm^3. With distilled water. Prepare 0.5 M oxalic acid by dissolving 31.5 gms of it in 250 cm^3. Of solution. First of all prepare standard solution of IM NaOH by it and then with standard IM HCI, prepare IM NaOH of exact molarity.

Take a calorimeter and wash it with stirrer. Weigh this calorimeter on a rough balance. Take 50 cm^3 of IM NaOH in it. Note the initial temperature of NaOH with the help of a thermometer.

Add 50 cm^3 of IM HCI in it. There will be a rise in temperature when acid and alkali react together. Note the rise in temperature of the mixture when the temperature stops rising, weigh the calorimeter and mixture solution. Find out the mass of the solution. Calculate the heat of neutralization as follow:





observations and Calculations:

(1) Weight of calorimeter + stirrer + lid = A_1 gram = m_1g.
(2) Weight of calorimeter + stirrer + lid + solution = A_2 grams
(3) Weight of solution = A_2-A_1 = m_2 grams
(4) Initial temperature = t_1°C
(5) Final temperature = t_2°C
(6) Rise in temperature = t_2-t_1 =°C = θ
(7) Specific heat of copper calorimeter = S_1 =0.3807J/gK or J/g C.
(8) Specific heat of dilute solution = S2 =4.184J/gK or J/g°C.
(9) V = Volume of acid or base = 50cm^3.
(10) M = Molarity of acid or base = 1M.

Formula:

$$\textbf{Heat of Neutralization} = \frac{(m_1 s_1 + m_2 s_2)\,\theta}{1000} \times \frac{1000}{V \times M} = x\text{KJ/mol.}$$

Result:

The heat of neutralization of NaOH and HCl is = –x kJ/mol.

COMMON ION EFFECT

Experiment:

To purify the given sample of commercial NaCl by passing HCl gas.

Principle:

(decreased)

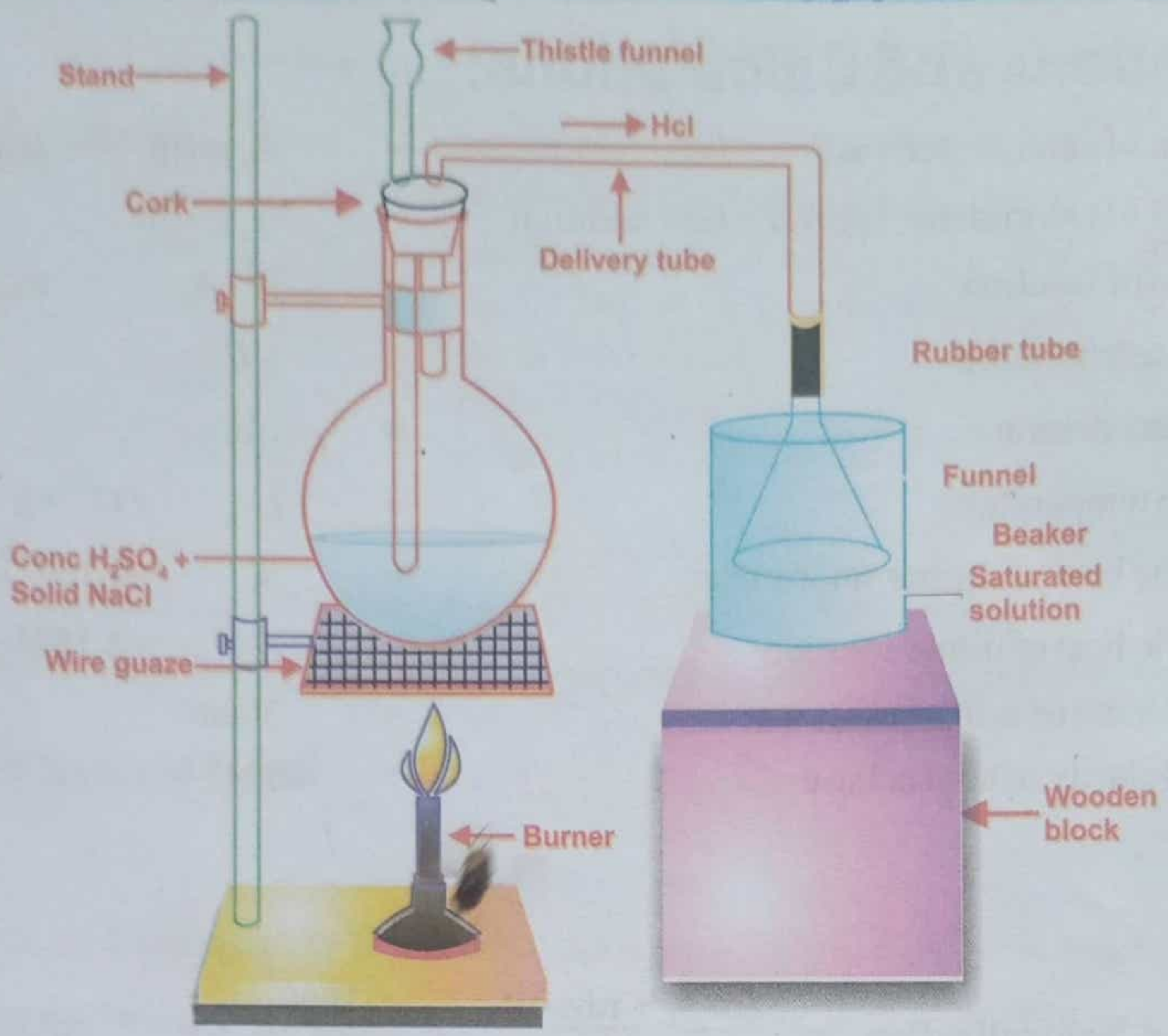
"The degree of ionization of an electrolyte is suppressed by the addition of another electrolyte having common ion is called common ion effect"

Procedure:

When we have a saturated solution of commercial NaCl then an equilibrium is established between ionized and solid NaCl at saturation as follows.

$$NaCl_{(s)} \rightleftharpoons Na^+_{(aq)} + Cl^-_{(aq)}$$

Now if a common ion e.g., Cl^- added in this solution by passing HCl gas, the molar concentration of Cl^- ions increases and as a result Na^+ ions and Cl^- ions combined to form solid NaCl. Take NaCl in the round bottom flask and add conc. H_2SO_4 through thistle funnel in it. When H_2SO_4 reacts with NaCl solid, a colourless punget gas HCl will evolve which passes through the delivery tube into the beaker containing saturated solution of NaCl. White crystals of pure NaCl will be precipitated out and settle down at the bottom. When no more crystals are formed, stop passing HCl gas. Separate the crystals of NaCl by filteration and dry them between folds of filter paper or in desiccator and show to the examiner.



Result:

Commercial NaCl is purified by passing HCl gas and crystals are shown to the examiner.

CRYSTALLIZATION:

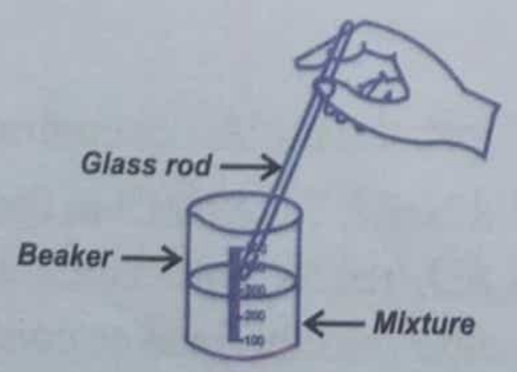
Experiment :

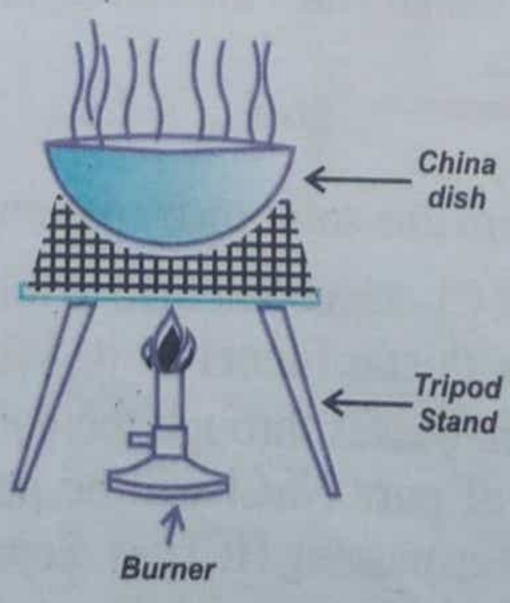
To prepare the crystals of benzoic acid from water.

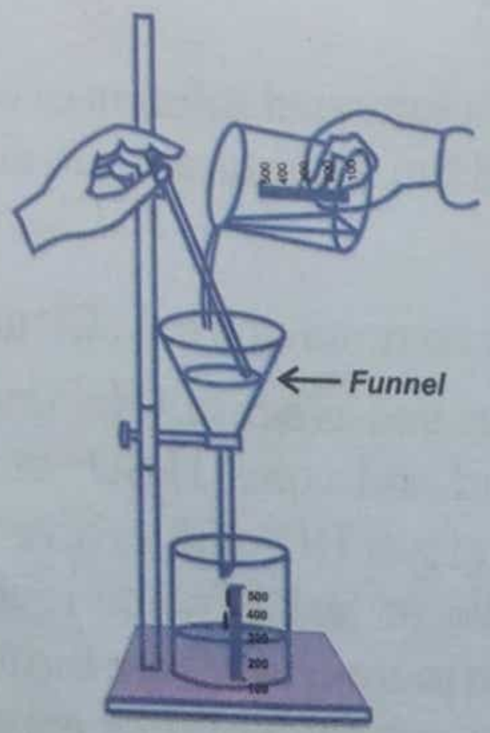
Principle:

"When hot super saturated solution is cooled at room temperature, solid crystals of benzoic acid are formed."

Benzoic acid is slightly soluble in water at room temperature ($0.5 g/dm^3$). However appreciable amount is soluble at high temperature, so when the temperature is lowered crystals are formed.







Apparatus:

Beaker, glass rod, china dish, tripod stand, stirrer, filter paper, wire guaze.

Procedure:

Take 50 to 60cm^3 water in 250cm^3 beaker. Add some benzoic acid in it at room temperature when no more acid dissolves then, heat the solution. Add further amount of acid till it will dissolve. Now filter the hot solution of benzoic acid in china dish and cool it. Don't give rapid cooling by placing it in ice. Cool the filterate at room temperature. On cooling, white lustrous flakes type crystals of benzoic acid will separate out. Decant the mother liquour and dry the crystals on filter paper by placing in desiccator and show to the examiner.

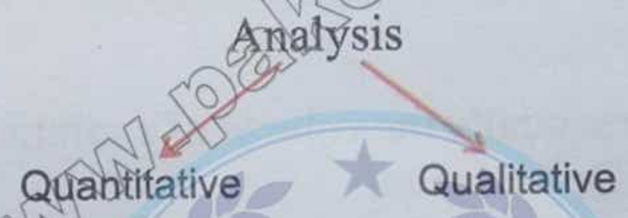
Result:

Crystals of benzoic and prepared and shown to the examiners.

PART-II
VOLUMETRIC ANALYSIS

Analysis:

Chemical characterization of compounds is called analysis.



Quantitative Analysis:

If the relative amounts of elements present in a compounds is determined then the analysis is called quantitative analysis.

Qualitative Analysis:

It is concerned with the detection or identification of elements present in a compound.e.g. defection of glucose as it contains carbon, hydrogen and oxygen involves qualitative analysis. We determine quality of chemicals.

Volumetric Analysis:

It is the branch of quantitative analysis in which volume of the reacting substances is to be measured. It has following applications. We can easily determine.

(1) Concentration of the solutions.
(2) Percentage composition of a mixture.
(3) Percentage purity of a substance.
(4) Number of water molecules in a crystalline hydrated solids.
(5) %age oxidation of partially oxidized sample.
(6) Solubility of different substances at room temperaure.
(7) Molecular and atomic weights of acids or bases.

observations and Calculations:

(1) Weight of calorimeter + stirrer + lid = A_1 gram = m_1g.

(2) Weight of calorimeter + stirrer + lid + solution = A_2 grams

(3) Weight of solution = A_2 - A_1 = m_2 grams

(4) Initial temperature = t_1°C

(5) Final temperature = t_2°C

(6) Rise in temperature = t_2-t_1 =°C = θ

(7) Specific heat of copper calorimeter = S_1 =0.3807J/gK or J/g C.

(8) Specific heat of dilute solution = S2 =4.184J/gK or J/g°C.

(9) V = Volume of acid or base = 50cm³.

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Formula:

$$\textbf{Heat of Neutralization} = \frac{(m_1s_1 + m_2s_2)\theta}{1000} \times \frac{1000}{V \times M} = x\text{KJ/mol.}$$

Result:

The heat of neutralization of NaOH and HCl is = –x kJ/mol.

COMMON ION EFFECT

Experiment:

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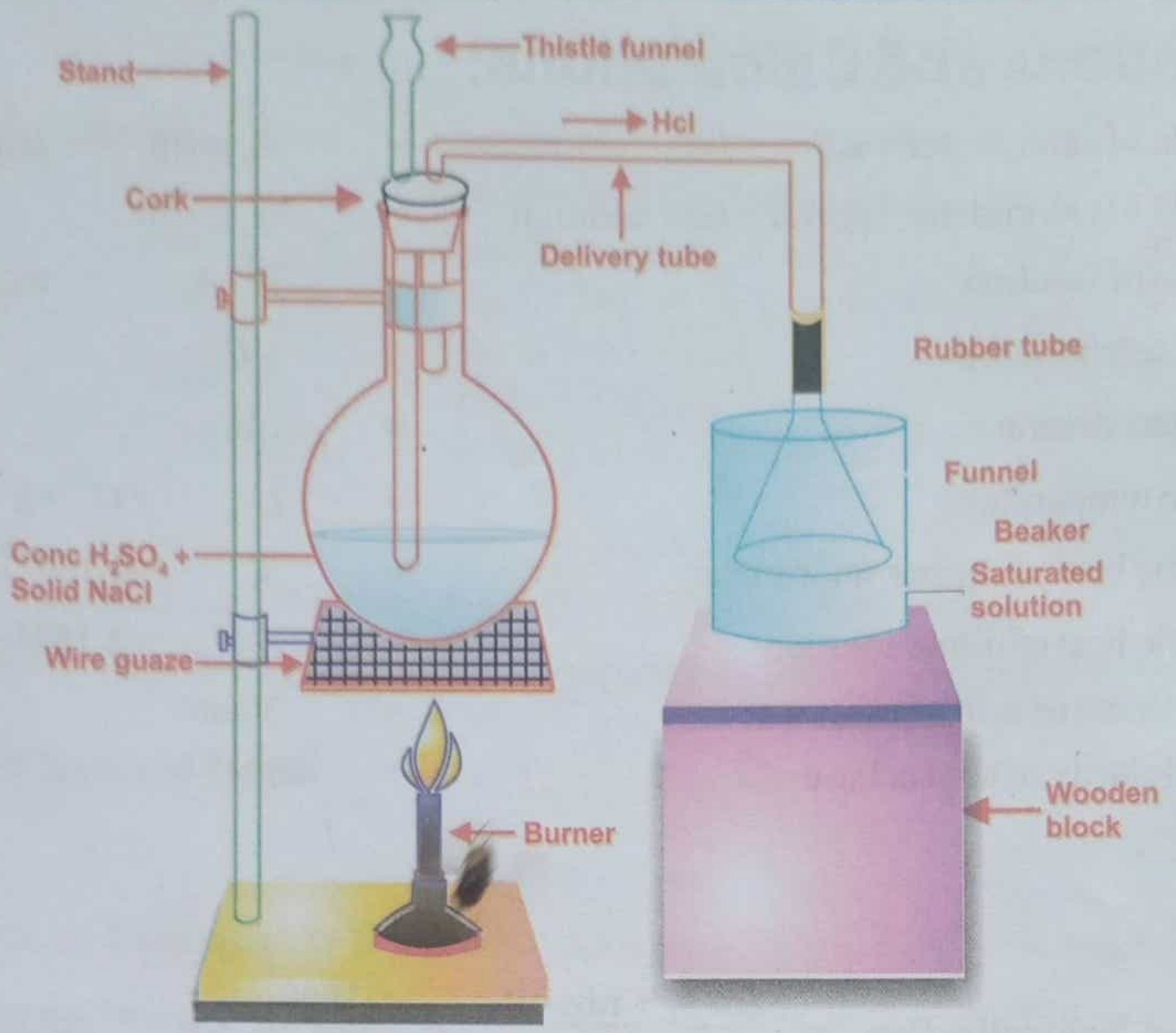


Result:

Commercial NaCl is purified by passing HCl gas and crystals are shown to the examiner.

CRYSTALLIZATION:

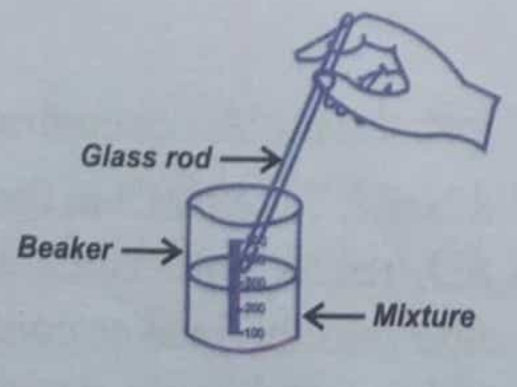
Experiment :

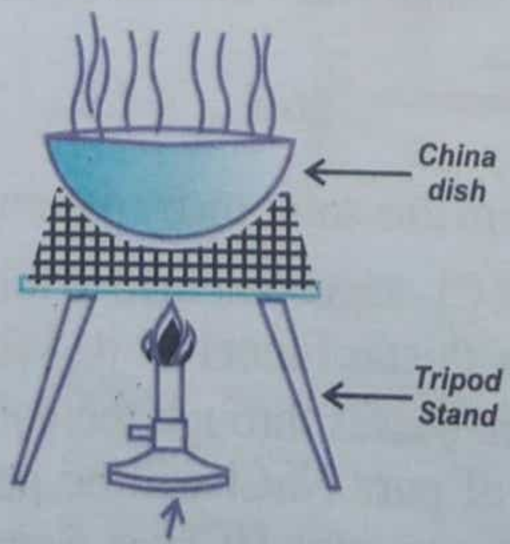
To prepare the crystals of benzoic acid from water.

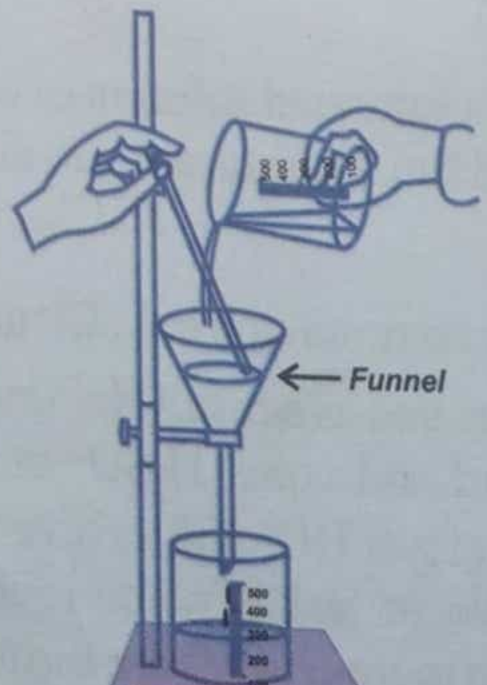
Principle:

"When hot super saturated solution is cooled at room temperature, solid crystals of benzoic acid are formed."

Benzoic acid is slightly soluble in water at room temperature ($0.5 g/dm^3$). However appreciable amount is soluble at high temperature, so when the temperature is lowered crystals are formed.







STUDY THE WORKING OF BUNSEN BURNER:

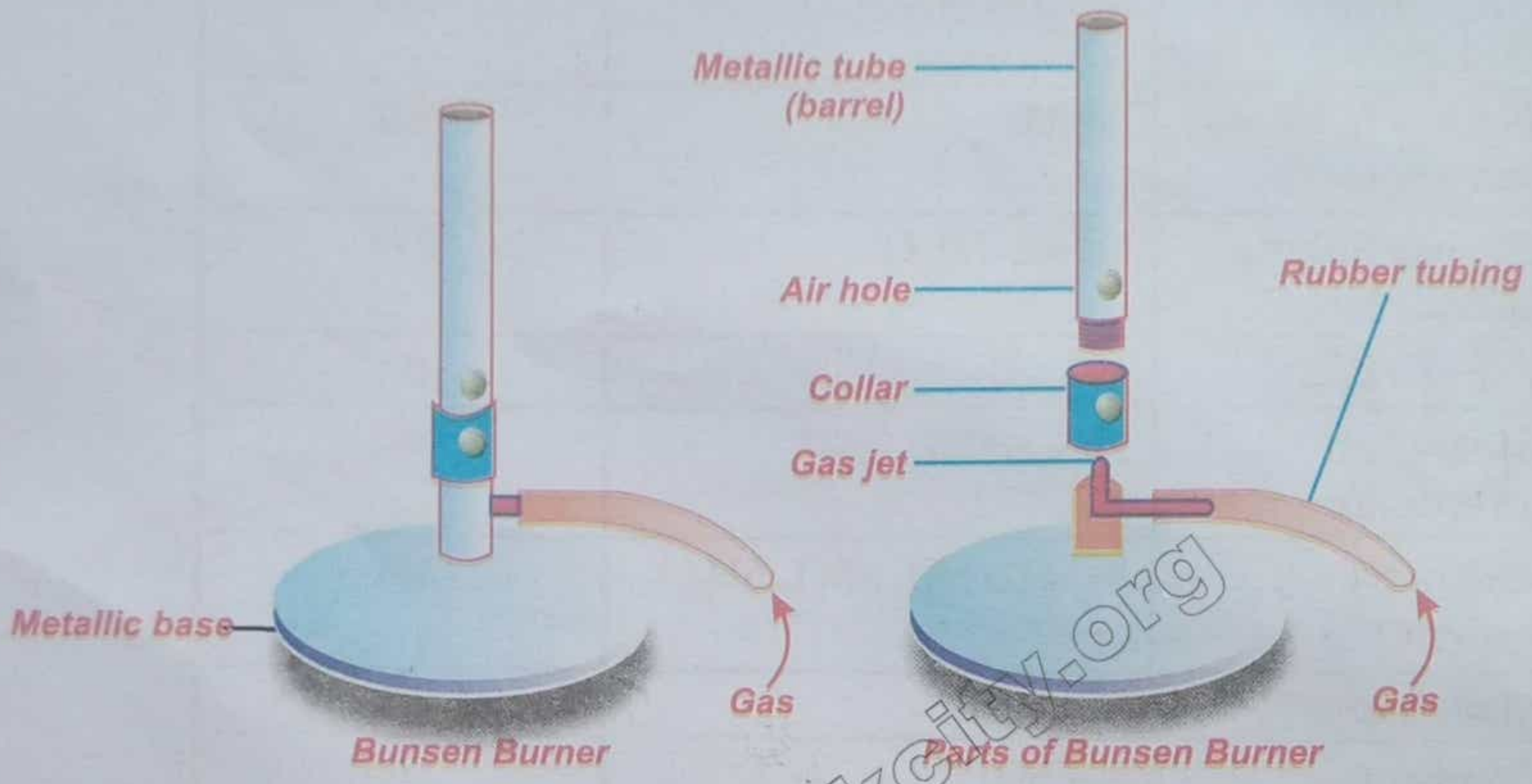
In Laboratories, we usually use Bunsen burner for heating purpose. It produce heat energy by burning of suigas, oil gas, kerosene or petrol gas in the laboratory.

PARTS OF BUNSEN BURNER:

It consist of the four parts.

(1) Metallic Base:

It is made of iron or steel and is the lowest supporting portion of the burner. The base contains a hole of the entry of gas.



Bunsen Burner

Parts of Bunsen Burner

PARTS OF BUNSEN BURNER

Nipple or the gas jet:

It is also made of iron or steel and has a fine hole through which gas comes out. It is fitted at upper end of the metallic base.

Metallic Tube:

It is a long cylindrical tube and is fitted on nipple. It is made of brass. It has two holes near the bottom to allow the air to enter and mix with the gas for complete combustion.

Air Regulator:

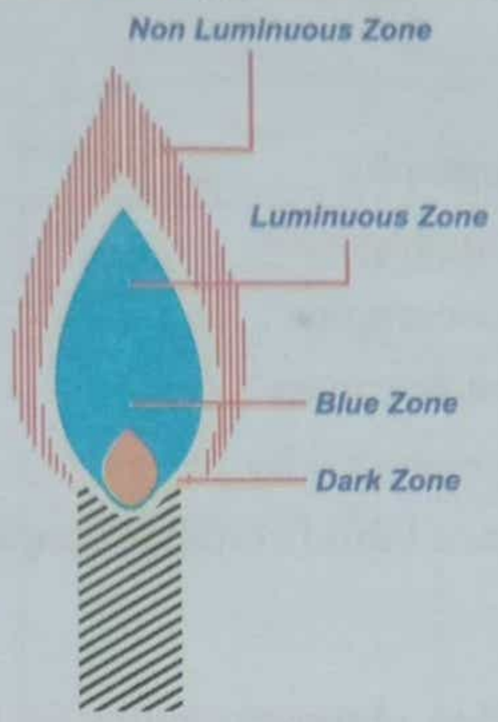
It is metallic ring which has two corresponding holes similar to the hole of tube. It regulates the quantity of air to get the flame of our choice whether oxidizing or reducing flame.

Parts of Flame:

Oxidizing or non-luminuos flame has the following parts.

The Dark Inner Zone:

It is the inner most zone of flame and is situated just above the burner tube. It is also called cold zone and does not possess heat or light. A match stick does not burn in it. It consists of unburnt particles of carbon.



PARTS OF FLAME

Blue Zone:

This is produced at the are of flame. This the hot part of flame. The colour of flame is blue in this zone due to burning of carbonmonoxide.

Luminous Zone:

It surrounds the dark inner zone. In this zone incomplete combustion takes place. This region contains mixture of CO and H_2. The zone is used for slow heating.

Non-Luminous Zone:

This is the outermost invisible zone of the flame. It is the hottest zone of flame since the combustion is complete in this zone. It surrounds the luminous zone.

CHROMATOGRAPHY:

It is an important application of distribution law. The word chromatography originates from the Greek word "Khromatos" measing colour and "graphy" mean writing or measurements.

It is non-destructive process for resolving a mutli-component mixture into its fractions. It is Quantitative as well as Qualitative analysis.

Distribution Law:

"A solute distributes itself between two immiscible liquids in a constant ratio of concentrations irrespective of the amount of solute added."

Principle

Chromatography:

Chromatography is a technique in which involves the distribution of components of mixture between stationary phase and a mobile phase.

In all chromatographic techniques, difference in affinity involves the process of either adsorption or partition.

Chromatography in which stationary phase is solid is called "adsorption chromatography.

Chromatography in which stationary phase and mobile phase both are liquids is called "partition chromatography."

The distribution of the components of a mixture between the two phases is governed by distribution coefficient 'K'.

$$K = \frac{\text{concentration of component in moving phase}}{\text{concentration of component in stationary phase}}$$

Principle

$$\text{or} \quad K = \frac{C_m}{C_s}$$

Kind Of Chromatography:

(1) Paper chromatography
(2) Thin layer chromatography (TLC)
(3) Column chromatography
(4) Gas-solid chromatography (GSC)
(5) Gas-liquid chromatography (GLC)
(6) High performance liquid chromatography (HPLC)

PAPER CHROMATOGRAPHY:

It is a technique of partition chromatography in which the stationary phase in water absorbed on paper and mobile phase is usually an organic liquid or mixture of liquids.

STATIONARY PHASE:

Some moisture held in paper and cellulose of paper acts as a stationary phase. In paper chromatography, filter paper serves as a support for this phase.

MOBILE PHASE:

It is the solvent which moves over the filter paper. The moving liquid may be immiscible or partially miscible with fixed phase.

PROCEDURE:

(i) Paper Preparation:

Take whatman filter paper no.1 and cut the paper in rectangular shape of about 7-8 width and 20cm height. (The choice of paper depends upon the porosity, thickness, purity and strength.

(ii) Sample Applications:

Make a base line with lead pencil on one edge of filter paper at one inch distance. Apply the sample with jet of capillary on base line. The spot should be minimum. Dry the spot and then put in the solvent.

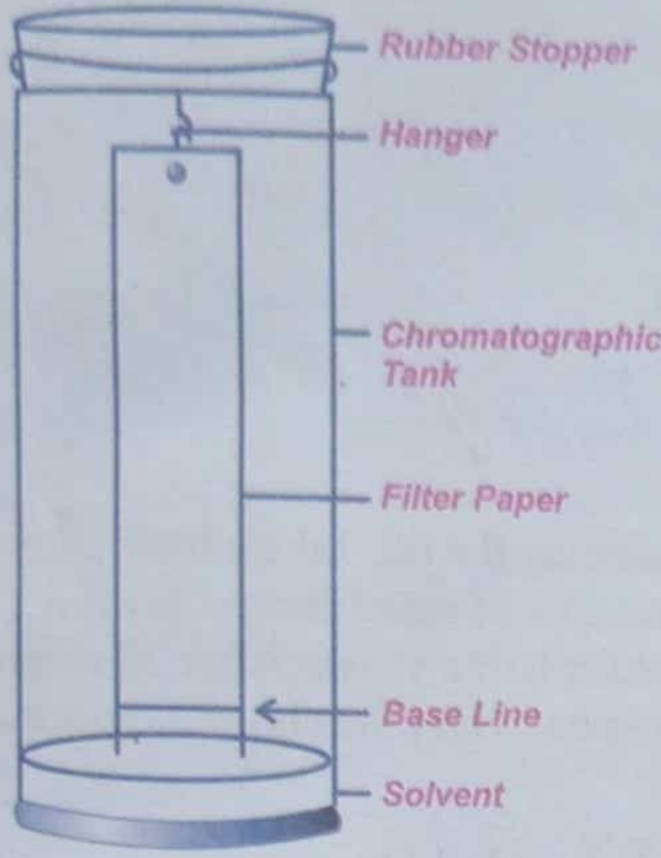
Choice of Solvent:

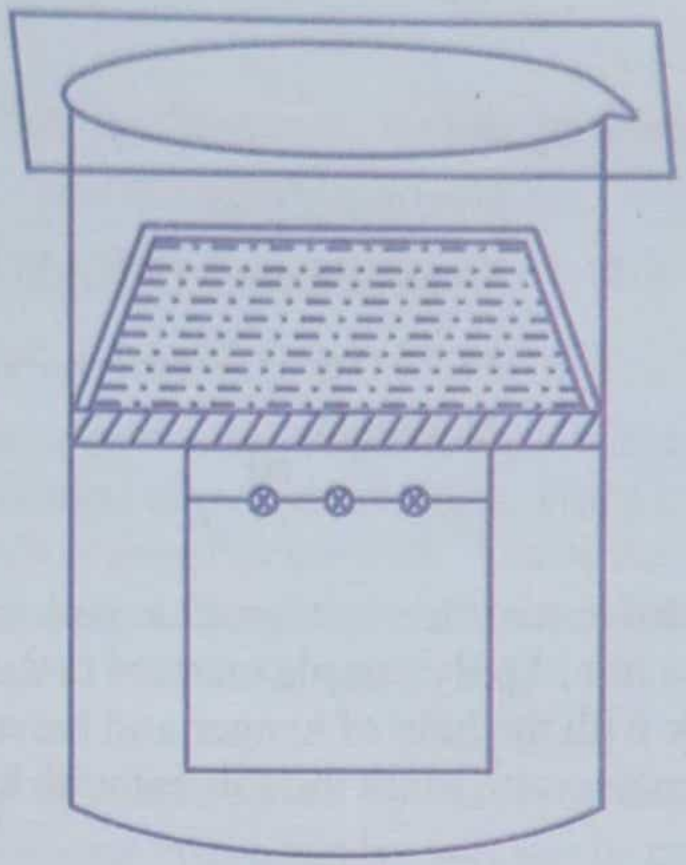
The choice of solvent depends upon the nature of substances to be separated. A single solvent or mixture of solvent is taken in chromatographic tank.

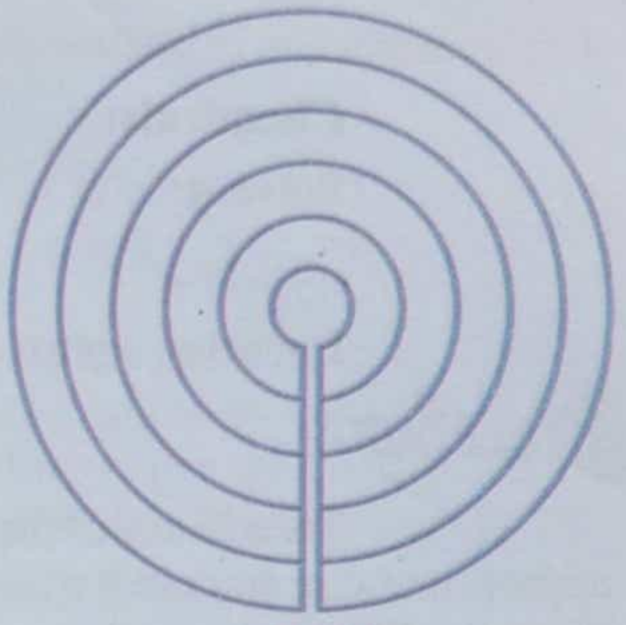
Development of Chromatogram:

Put the solvent in chromatographic tank and cover it for few minutes. Suspend the filter paper strip with the hanger in such a way that base line should be above the solvent and just the edge of paper should touch the solvent. Paper also should not touch the walls of tank.

In paper chromatography, development of chromatogram take place by ascending descending or radial ways.




Descending


Radial

Location of Spot:

If spots are coloured, no locating agent will be used. If spots are colourless, some locating agent must be used.

For location of Pb^{2+} and Cd^{2+} ions, H_2S can be used as locating agent.

1. Take some FeS pieces in beaker and add dilute H_2SO_4 and put into the desiccator. Put paper in the desiccator for sometime.
2. Take some pieces of Na_2S and mix it in water. Filter the solution and use the filterate as aqueous sodium sulphide solution which provides sulphide ions for location of lead and cadmium ions spots.
3. Some spraying agent can also be used. e.g., Ninhydrin, rubeanic acid, dithizone.

R_f Value:

R_f value expresses relative rates of movement of solute and solvent.

$$R_f = \frac{\text{distance travelled by solute in cm}}{\text{distance travelled by solvent in cm}}$$

R_f value is a ratio and has no unit. Its value is always less than one.

Experiment:

To separate the mixture of inks by paper chromatography:

/ **Chemicals:** Mixture of inks

Solvent: Water: Ethanol: $(NH_4)_2SO_4$

Mobile phase

$18.75\ cm^3 : 3.75 cm^3 : 2.5 cm^3$

Locating agent: Not required / Pencil page

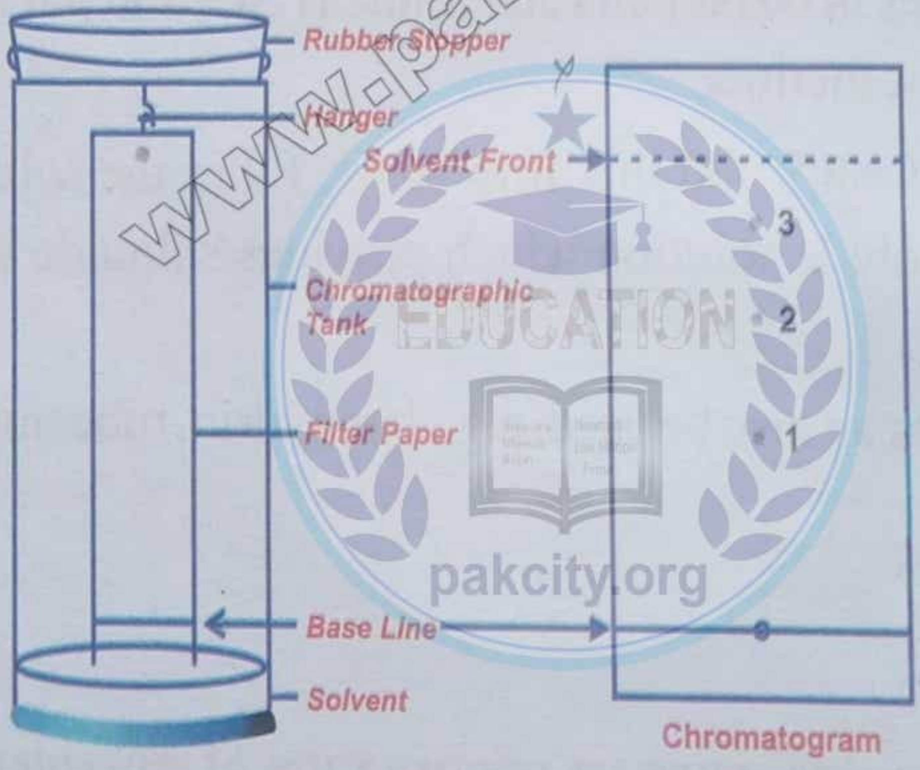
Procedure:

Take $25 cm^3$ of the solvent in chromatographic tank and cover the lid. Take a filter paper of appropriate size and mark a base line. Apply sample mixture in the middle of base line and dry the spot. Place the filter paper in the tank with the help of hanger and leave it for half an hour. After 30 minutes remove the lid and put out chromatogram. Mark the solvent with lead pencil Dry the chromatogram and calculate R_f values.

$$R_f = \frac{\text{Distance travelled by solute in cm's}}{\text{Distance travelled by solvent in cm's}}$$

B 0.5cm
R 1.2cm
G 6cm

Band	Distance of solutes (cm)	Distance of solvent front (cm)	R_f value.
1. Green	12	7.8	1.53
2. Red	12	3.6 cm	3.33
3. Blue	12	5.5 cm	2.4



Result:

The given mixture of inks is separated by paper chromatography.

Experiment:

To separate the mixture of lead and cadmium ions by paper chromatography:

Chemicals: Mixture of two metal ions (Lead nitrate and cadmium nitrate)

Solvent: Any one of the solvent can be used.

(1) Ethanol : Water : 1MHCl
$22.5cm^3$ 1.25 $1.25cm^3$

(2) Ethanol : 2M HCl
$88cm^3$ $12cm^3$

Experiment:

To separate the mixture of inks by paper chromatography:

Chemicals: Mixture of inks

Solvent: Water: Ethanol: $(NH_4)_2SO_4$

Mobile phase

$18.75\,cm^3 : 3.75\,cm^3 : 2.5\,cm^3$

Locating agent: Not required / Pencil page

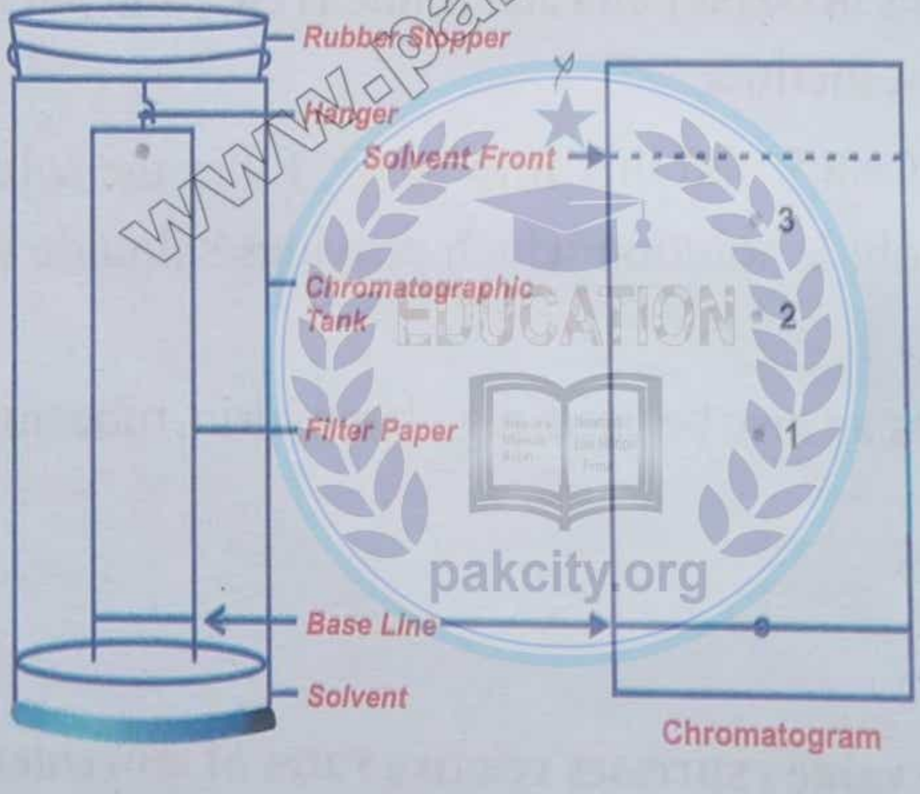
Procedure:

Take 25cm³ of the solvent in chromatographic tank and cover the lid. Take a filter paper of appropriate size and mark a base line. Apply sample mixture in the middle of base line and dry the spot. Place the filter paper in the tank with the help of hanger and leave it for half an hour. After 30 minutes remove the lid and put out chromatogram. Mark the solvent with lead pencil Dry the chromatogram and calculate R_f values.

$$R_f = \frac{\text{Distance travelled by solute in cm's}}{\text{Distance travelled by solvent in cm's}}$$

B 0.5cm
R 1.2cm
G 6cm

Band	Distance of solutes (cm)	Distance of solvent front (cm)	R_f value.
1. Green	12	7.8	1.53
2. Red	12	3.6 cm	3.33
3. Blue	12	0.5 cm	24



Result:

The given mixture of inks is separated by paper chromatography.

Experiment:

To separate the mixture of lead and cadmium ions by paper chromatography:

Chemicals: Mixture of two metal ions (Lead nitrate and cadmium nitrate)

Solvent: Any one of the solvent can be used.

(1) Ethanol : Water : 1MHCl
22.5cm³ 1.25 1.25cm³

(2) Ethanol : 2M HCl
88cm³ 12cm³

Locating Agent:

(1) H_2S, Cadmium gives yellow and plumbous give brownish black spot.

(2) 5% KI spray. Both Pb^{2+} and Cd^{2+} gives yellow spots.

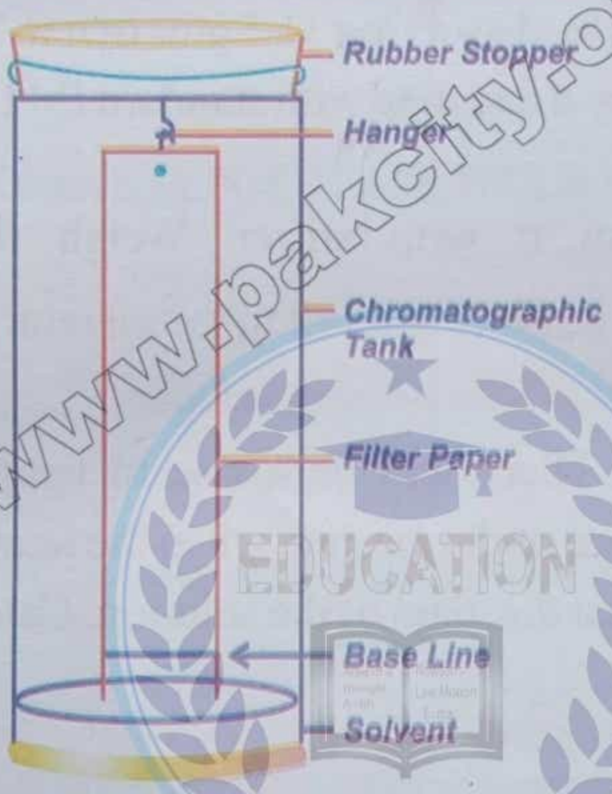
(3) 0.05% dithizone in CCl_4 spray Pb^{2+} gives red spot while Cd^{2+} give pink spot

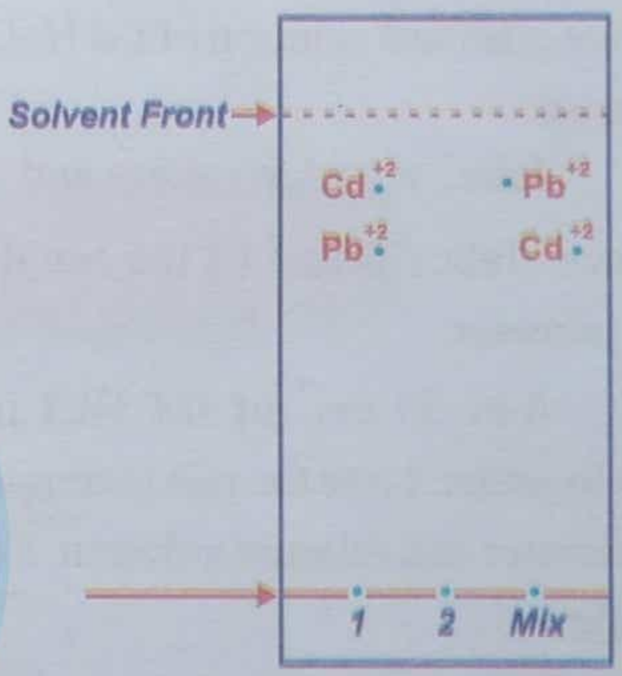
Procedure:

Take the solvent in chromatographic tank and cover it. Take a filter paper strip of appropriate size and mark a base line on one edge of paper. Place a spot of sample mixture in the middle of base-line and hang the filter paper in the tank. Leave the filter paper for 1½hr. After 1½hr, remove the lid and dry the chromatogram. Since the spots are colourless, so locating agent is sprayed on strip. Two spot yellow and brown appeared on the strip. Dry the chromatogram and calculate the Rf value for both ions.

$$R_f = \frac{\text{Distance travelled by solute in cm's}}{\text{Distance travelled by solvent in cm's}}$$

	Ions	Distance of solute in cm	Distance of solvent front in cm	R_f value
Brow	Lead	11.5	9 cm	1.278
Yellow	Cadmium	11.5	4.5 cm	2.56





Result:

The given mixture of Pb^{2+} and Cd^{2+} ions is separated by chromatography.

Heat of Neutralization:

Neutralization:

"The process in which an acid reacts with a base to form salt and water is called neutralization".

When NaOH reacts with HCl, NaCl and H_2O is formed as follows.

$$NaOH + HCl \longrightarrow NaCl + H_2O$$
$$OH^- + H_3O^+ \longrightarrow 2H_2O$$

Heat of Neutralization:

The amount of heat evolved when one mole of water is formed by complete neutralization of an acid with base is called heat of neutralization. e.g.

$$HCl_{(aq)} + NaOH_{aq} \longrightarrow NaCl + H_2O$$
$$H^+ + \bar{O}H \longrightarrow H_2O \quad (\Delta H = -57.32kJ)$$

It is an exothermic process and heat is evolved when a strong acid neutralizes a strong base.

$$\text{Heat of neutralization} = \frac{(m_1s_1 + m_2s_2)\,\theta}{1000} \times \frac{1000}{V \times M}$$

Where m_1 = mass of calorimeter
s_1 = specific heat of calorimeter
m_2 = mass of solution
s_2 = specific heat of dilute solution
θ = Rise in temperature
V = volume of acid or base used
M = Molarity of acid or base used

Experiment:

Determine the Heat of Neutralization of a Strong Acid with Strong Base:

Apparatus: ***Calorimeter, Beakers, thermometer, weight box, balance stopwatch measuring cylinder.***

Chemical: **1M HCl, 1M NaOH**

Procedure:

For preparing approximately IM NaOH, take 4 gm of it, and dissolve it in 100 cm^3 of solution and for preparing approximately IM HCI, take 10 cm^3 of conc. HCI and dilute it to 100 cm^3. With distilled water. Prepare 0.5 M oxalic acid by dissolving 31.5 gms of it in 250 cm^3. Of solution. First of all prepare standard solution of IM NaOH by it and then with standard IM HCI, prepare IM NaOH of exact molarity.

Take a calorimeter and wash it with stirrer. Weigh this calorimeter on a rough balance. Take 50 cm^3 of IM NaOH in it. Note the initial temperature of NaOH with the help of a thermometer.

Add 50 cm^3 of IM HCI in it. There will be a rise in temperature when acid and alkali react together. Note the rise in temperature of the mixture when the temperature stops rising, weigh the calorimeter and mixture solution. Find out the mass of the solution. Calculate the heat of neutralization as follow: