DIRECTORATE OF DISTANCE & CONTINUING EDUCATIONS

MANONMANIAM SUNDARANAR UNIVERSITY

TIRUNELVELI – 627012

OPEN AND DISTANCE LEARING(ODL) PROGRAMMES

(FOR THOSE WHO JOINED THE PROGRMMES FROM THE ACADEMIC YEAR 2023 – 2024)

M.Sc. CHEMSITY
COURSE MATERIALS
PREPARATION OF ORGANIC AND INORGANIC COMPOUNDS AND PHYSICAL CONSTANT
JMCHP4

By

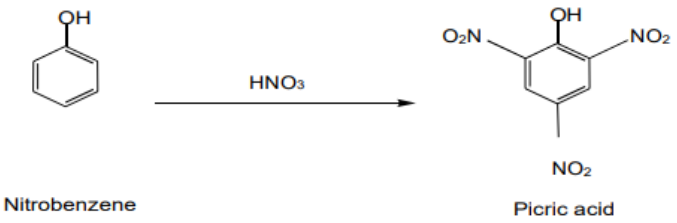
S. NAGARAJAN
Assistant Professor
Department of Chemistry
Manonmaniam Sundaranar University
Tirunelveli 627012

UNIT 1

Preparation of Organic Compounds

i) Nitration-picric acid from Phenol

Picric acid is 2,4,6-trinitro-phenol. It is yellow in colour. It is used as tropical anti-infective and disinfectant so that used as a cleaning agent and also preservative due to presence of phenol. Picric acid is obtained by nitrating phenol. In pharmacology experiments, picric acid is also used to mark the rats and mice. Nitration: Nitration is an example of electrophilic aromatic substitution reaction. A large no. of aromatic compounds can be easily nitrated. The hydrogen atom replaced by nitro group. The nitration of aromatic compounds is usually done by using conc. HNO_3 in presence of conc. H_2SO_4. Nitration of aromatic compounds is an example of electrophilic aromatic substitution. H_2SO_4 not only provides strong acidic medium but it also converts the HNO_3 into reactive electrophile nitronium ion (NO^{+2}) which attacks the aromatic ring. Nitration is usually carried out at low temperature. At high temperature there is loss of material due to oxidation by HNO_3. Phenol being an activated nucleus towards electrophilic aromatic substitution, the nitration reaction occurs very easily. It undergoes nitration with HNO3 even at room temperature forming ortho & para nitrophenol which can be separated by steam distillation. Phenol when treated with conc. HNO_3 in presence of conc. H_2SO_4 undergoes nitration at both ortho and para position to yield picric acid. It is better if phenol is first converted into phenol sulphonic acid by treatment with H_2SO_4 and then nitrated with conc. HNO_3.



Nitrobenzene Picric acid

PROCEDURE:

Take 4gm/mL of phenol into a dry 250 mL of conical flak. Add 5 mL of conc. H_2SO_4 & mix thoroughly which becomes warm because the reaction is exothermic, now heat the flask on the boiling water bath for 30 mins to complete the formation of phenol sulphonic acid & then chill the flask thoroughly in ice water mixture. Place the flask on a wooden block, add immediately 15 mL of conc. HNO_3 and at once mix the liquid by shaking for few minutes. Then allow the mixture to stand undisturbed. Usually within 1 min a vigorous but harmless reaction occurs

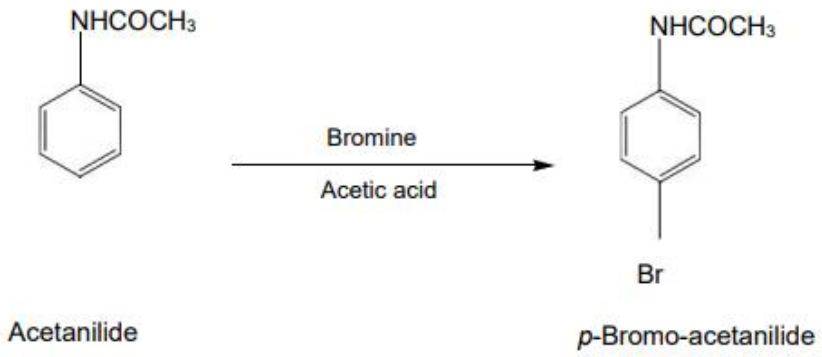
and red fumes pour out of the flask when the action subsides, heat the flask on boiling water bath for 1-2 hrs with occasional stirring. During this period, the heavy oils which is present at the beginning ultimately forms a mass of crystals. Add 100 mL of cold water and then chill thoroughly, mixing well. Filter the yellow crystals wash thoroughly with water to eliminate all inorganic acids & drain.

2. Halo genation-p-bromoacet anilide from acetanilide

Halogenation of aromatic compound is an e.g. of electrophillic aromatic substitution reaction i.e. chlorination or bromination of benzene. The major role of halogen carriers is to generate a brominium ion electrophile that eventually attacks the nucleus at the particular site of maximum electron density. Acetanilide can be easily brominated with bromine in glacial acetic acid. In this reaction small amount of o–bromo acetanilide is also formed which being more soluble in alcohol can be removed during crystallization of para bromo acetanilide with the liberation of a mole of hydrogen bromide (-$NHCOCH_3$) is an ortho, para directing function. Hence the incoming bromo mostly shall yield both ortho & para isomers. The later is produced predominately up to 90% as a white solid.

PROCEDURE:-

Take 1 gm finely powdered acetanilide in 5 mL cold glacial acetic acid containing 250 mL of conical flask. Chill the content of conical flask then immediately add 6.5 mL of bromine solution from the burette drop wise very slowly with vigorous shaking in chilled content of conical flask. After mixing & shaking, allow the mixture to stand at room temperature for 25 minutes. Then pour the pale reddish orange solution which may contain some crystal of para-bromo-acetanilide into a large excess amount i.e. about 60 mL of cold water where upon the para-bromo-acetanilide will rapidly crystallize out. Stir these crystals thoroughly & wash with the water to eliminate acetic acid, unchanged bromine etc. & then filter, wash well with cold water, drain & finally Recrystallize. The melting point is about 167o C & yield is about 1 gm. The crude product may be recrystallized from the rectified spirit either at room temperature or slightly warming it in electric water bath. The yield of pure coloured para-bromo-acetanilide is slightly reduced.

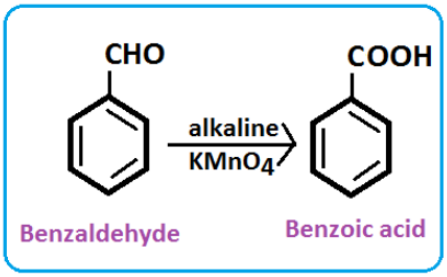


3. Oxidation-benzoic acid from Benzaldehyde

Oxidation of aldehydes to carboxylic acids is an example of a redox reaction, where the aldehyde (-CHO) functional group is converted into a carboxyl (-COOH) group by an oxidizing agent. Benzaldehyde undergoes oxidation to form benzoic acid using potassium permanganate ($KMnO_4$) in an alkaline medium. The reaction initially forms potassium benzoate, which upon acidification, yields benzoic acid as a white crystalline solid. A small amount of side products, such as manganese dioxide (MnO_2), may be formed, which are removed by filtration.

Procedure:

In a 250 mL conical flask, 2 mL of benzaldehyde is mixed with 50 mL of distilled water. A solution of 1 g potassium permanganate ($KMnO_4$) in 10 mL water is prepared and added dropwise to the reaction mixture with constant stirring. To maintain an alkaline medium, a few drops of NaOH solution are added. The mixture is then gently heated on a water bath at 60–70°C for approximately 30 minutes, during which the purple color of $KMnO_4$ gradually disappears, indicating the completion of the oxidation process. The reaction mixture is cooled, and the brown precipitate of MnO_2 formed is removed by filtration. The clear filtrate is then acidified by adding dilute HCl or H_2SO_4 dropwise until the pH reaches 2–3, leading to the precipitation of benzoic acid as a white solid. The solid is filtered, washed with cold water to remove any impurities, and dried. The crude product is then recrystallized using hot water to obtain purified benzoic acid.



Results:

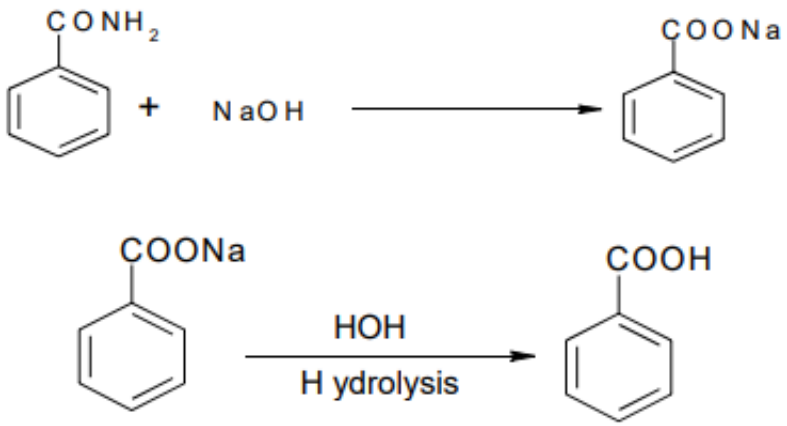
The oxidation of benzaldehyde using potassium permanganate yielded pure benzoic acid as a white crystalline solid. The melting point of the purified benzoic acid was found to be 121–123°C, confirming its purity. The expected yield was approximately 1 g, with minimal impurities due to proper recrystallization.

4. Benzoic acid from Benzamide

Benzoic acid is simplest aromatic carboxylic acid. It is used for the preparation of various important compounds. Itself it acts as keratolytic and antifungal agent.

PROCEDURE: -

Placed the mixture of conc. NH_3 (10 mL) and water (5 mL) in a conical flask & shake vigorously and add benzoyl chloride drop by drop formed the benzamide as separated. Take 1 gm of formed benzamide & 10 % NaOH (15 mL) in a RBF flask fitted with refluxes condenses and boil the mixture gently 30 min. ammonia is evolved then add con. H_2SO_4 after cooling the mixture until it becomes acidic ad benzoic acid is immediately separated out.

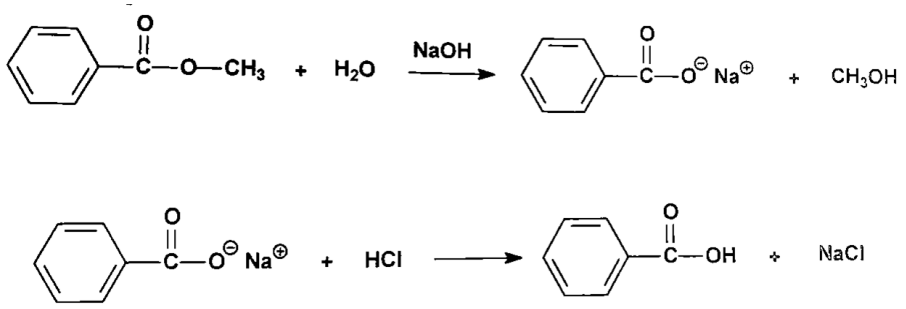


5. Methyl benzoate to Benzoic acid

The hydrolysis of methyl benzoate to benzoic acid is an example of an ester hydrolysis reaction. This reaction occurs in either acidic or basic medium, where methyl benzoate undergoes cleavage to yield benzoic acid. In alkaline hydrolysis (saponification), methyl benzoate is treated with aqueous sodium hydroxide (NaOH), forming sodium benzoate as an intermediate. Upon acidification, benzoic acid precipitates out as a white crystalline solid.

Procedure:

In a 250 mL conical flask, 2 mL of methyl benzoate is mixed with 25 mL of 10% sodium hydroxide (NaOH) solution. The mixture is heated under gentle reflux for about 30 minutes until complete hydrolysis occurs. After heating, the reaction mixture is cooled to room temperature and transferred into a beaker containing 50 mL of cold water. The solution is then acidified by adding dilute hydrochloric acid (HCl) dropwise, until the pH reaches 2–3, leading to the precipitation of benzoic acid. The precipitated solid is filtered using vacuum filtration, washed with cold water, and dried. To purify the product, the crude benzoic acid is recrystallized from hot water to obtain pure white crystals.



Results:

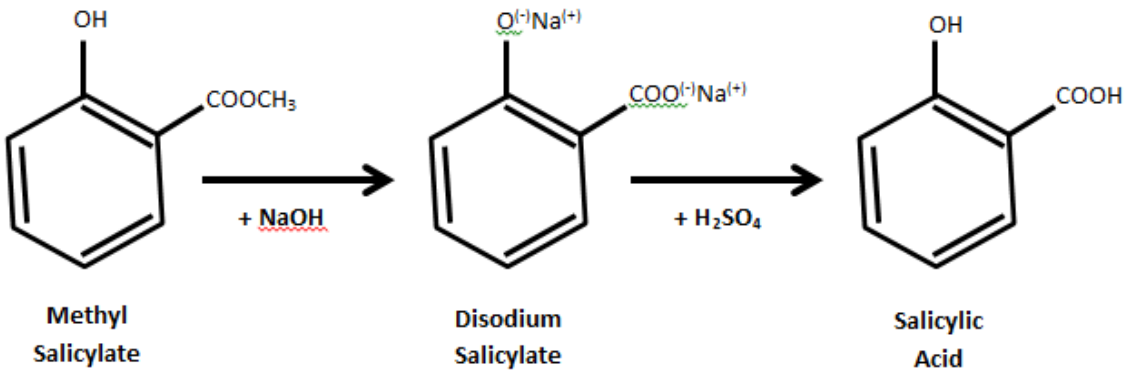
The alkaline hydrolysis of methyl benzoate successfully yielded benzoic acid as a white crystalline solid. The melting point of the purified benzoic acid was recorded as 121–123°C, confirming its purity. The expected yield was approximately 1 g, with minimal impurities due to effective recrystallization.

6. Salicylic acid from Methyl Salicylate

The hydrolysis of methyl salicylate to salicylic acid is an example of an ester hydrolysis reaction, where the ester functional group (-$COOCH_3$) undergoes cleavage in the presence of a strong base. The reaction occurs via alkaline hydrolysis (saponification) using sodium hydroxide (NaOH), forming sodium salicylate as an intermediate. Upon acidification, salicylic acid precipitates as a white crystalline solid.

Procedure:

In a 250 mL conical flask, 2 mL of methyl salicylate is mixed with 25 mL of 10% sodium hydroxide (NaOH) solution. The mixture is gently heated under reflux for about 30 minutes, ensuring complete hydrolysis. After cooling the reaction mixture to room temperature, it is acidified by adding H_2SO_4 dropwise, until the pH reaches 2–3, leading to the precipitation of salicylic acid. The solid is filtered using vacuum filtration, washed with cold water, and dried. To purify the product, the crude salicylic acid is recrystallized from hot water to obtain pure white crystals.



Results:

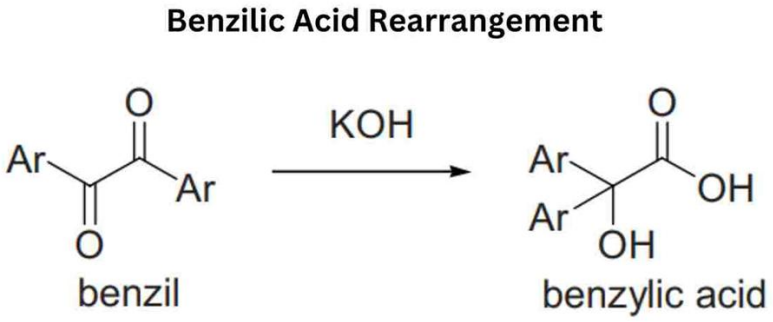
The hydrolysis of methyl salicylate successfully yielded salicylic acid as a white crystalline solid. The melting point of the purified salicylic acid was found to be 158–161°C, confirming its purity. The expected yield was approximately 1 g, with high purity achieved through recrystallization.

7. Rearrangement-Benzil to Benzilic Acid

The conversion of benzil to benzilic acid is an example of a benzilic acid rearrangement, a reaction where 1,2-diketones undergo rearrangement under basic conditions to form α-hydroxy acids. In this reaction, benzil is treated with potassium hydroxide (KOH), leading to rearrangement and the formation of potassium benzilate. Upon acidification, benzilic acid precipitates as a white crystalline solid.

Procedure:

In a 250 mL conical flask, dissolve 1 g of benzil in 10 mL of ethanol. To this solution, add 5 mL of 10% potassium hydroxide (KOH) solution while stirring continuously. The mixture is then gently heated under reflux for about 30 minutes, allowing the rearrangement to occur. After completion, the reaction mixture is cooled to room temperature and acidified with dilute hydrochloric acid (HCl) dropwise, until the pH reaches 2–3, leading to the precipitation of benzilic acid. The solid is filtered using vacuum filtration, washed with cold water, and dried. To purify the product, the crude benzilic acid is recrystallized from hot water to obtain pure white crystals.



Results:

The rearrangement of benzil successfully yielded benzilic acid as a white crystalline solid. The melting point of the purified benzilic acid was found to be 150–152°C, confirming its purity. The expected yield was approximately 1 g, with high purity achieved through recrystallization.

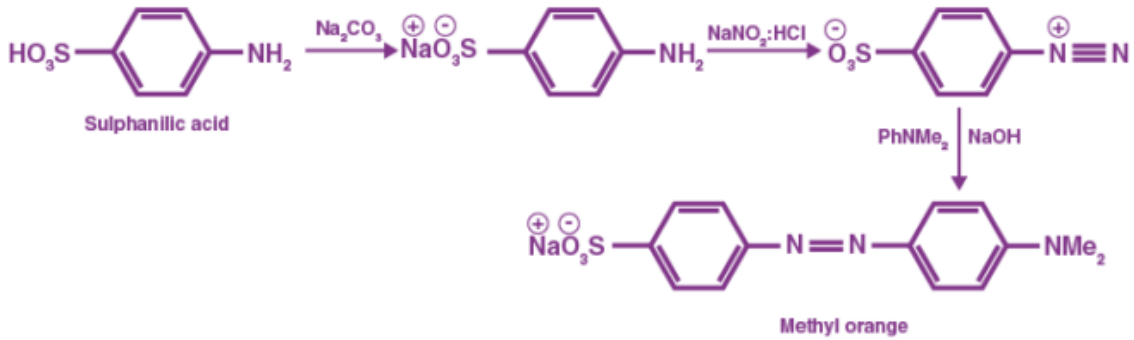
8. Methyl orange from sulphanilic acid

The synthesis of methyl orange from sulphanilic acid involves two main steps: diazotization and coupling reaction. In the first step, sulphanilic acid is diazotized using sodium nitrite ($NaNO_2$) in acidic medium to form a diazonium salt. In the second step, this diazonium salt is coupled with N,N-dimethylaniline, leading to the formation of methyl orange, a synthetic azo dye.

Procedure:

In a 250 mL beaker, dissolve 1 g of sulphanilic acid in 10 mL of 2N hydrochloric acid (HCl) and cool the solution to 0–5°C using an ice bath. In a separate beaker, prepare a solution of 0.5 g sodium nitrite ($NaNO_2$) in 5 mL of water. Add this sodium nitrite solution dropwise to the cold sulphanilic acid solution while stirring continuously. The formation of a diazonium salt is confirmed when a clear solution appears.

In another beaker, prepare a solution of 1 g of N,N-dimethylaniline in 5 mL of glacial acetic acid. Cool this solution and then slowly add the diazonium salt solution dropwise with constant stirring. The reaction mixture is allowed to stand for 15–20 minutes, leading to the formation of methyl orange as a precipitate. The solid product is filtered using vacuum filtration, washed with cold water, and dried. The crude methyl orange is then purified by recrystallization using ethanol.



Results:

Methyl orange was successfully synthesized as an orange-colored crystalline solid. The yield was approximately 1 g, and the purity was confirmed by its melting point (≥250°C,

decomposition). The final product can be used as a pH indicator, showing a color transition from red in acidic medium to yellow in basic medium.

UNIT II

Preparation of Inorganic Compounds

i. Potash alum

Potash alum, also known as potassium aluminium sulfate, is a double salt of potassium sulfate (K_2SO_4) and aluminium sulfate ($Al_2(SO_4)_3$). It is widely used in water purification, dyeing, and as an astringent. The synthesis involves the reaction of aluminium sulfate and potassium sulfate in water, followed by crystallization to obtain pure potash alum.

Procedure:

In a 250 mL beaker, dissolve 10 g of aluminium sulfate ($Al_2(SO_4)_3 \cdot 18H_2O$) in 50 mL of distilled water by heating and stirring gently. In another beaker, dissolve 2 g of potassium sulfate (K_2SO_4) in 20 mL of distilled water. Slowly add the potassium sulfate solution to the aluminium sulfate solution with continuous stirring. Heat the mixture to 60–70°C and allow it to dissolve completely. After dissolution, cool the solution to room temperature and then place it in an ice bath for crystallization. Shiny, colourless crystals of potash alum start forming after a few hours. The crystals are collected by vacuum filtration, washed with cold distilled water, and dried at room temperature.

$$Al_2(SO_4)_3 \cdot 18H_2O + K_2SO_4 + 6H_2O \rightarrow K_2SO_4 \cdot Al_2(SO_4)_3 \cdot 24H_2O$$

Results:

Potash alum was successfully synthesized as colourless, transparent octahedral crystals. The yield was approximately 80–90% of the theoretical amount. The product was confirmed by its solubility in water, acidic nature, and characteristic melting point of 92°C.

ii. Tetraamine copper (II) sulphate

Tetraamine copper(II) sulphate $[Cu(NH_3)_4]SO_4 \cdot H_2O[Cu(NH_3)_4]SO_4 \cdot H_2O[Cu(NH_3)_4]SO_4 \cdot H_2O$ is a coordination complex formed by the reaction of copper(II) sulfate with ammonia. It appears as deep blue crystals and is commonly used in coordination chemistry studies.

Procedure:

In a 250 mL beaker, dissolve 5 g of copper (II) sulfate pentahydrate ($CuSO_4 \cdot 5H_2O$) in 20 mL of distilled water with gentle stirring. Slowly add concentrated ammonia solution (NH_3, 25%) dropwise while stirring until a deep blue solution is obtained. The solution is then cooled in an ice bath, and ethanol (20 mL) is added slowly to initiate crystallization. The beaker is left undisturbed for 30 minutes, allowing deep blue tetraamine copper(II) sulfate crystals to form. The crystals are collected using vacuum filtration, washed with cold ethanol, and dried in air.

$$CuSO_4 \cdot 5H_2O + 4NH_3 \rightarrow [Cu(NH_3)_4]SO_4 \cdot H_2O + 4H_2O$$

Results:

Tetraamine copper(II) sulfate was successfully synthesized as deep blue crystalline solid. The expected yield was approximately 80–90%, and the presence of the complex was confirmed by its characteristic deep blue color and solubility in water.

iii. Hexamine cobalt (III) chloride

Hexamine cobalt(III) chloride is a coordination complex in which cobalt(III) is surrounded by six ammonia molecules as ligands. It is a stable, orange-yellow crystalline compound, commonly used in coordination chemistry and catalysis.

Procedure:

In a 250 mL beaker, dissolve 2.5 g of cobalt (II) chloride hexahydrate ($CoCl_2 \cdot 6H_2O$) in 20 mL of distilled water with gentle stirring. Add 10 mL of concentrated ammonia solution (NH_3, 25%) slowly while stirring, leading to the formation of a deep blue solution due to the formation of the hexamine cobalt (II) complex. Next, add 1 g of ammonium chloride (NH_4Cl) to stabilize the complex.

To oxidize cobalt (II) to cobalt (III), add 5 mL of 3% hydrogen peroxide (H_2O_2) dropwise while maintaining the solution at 50–60°C. The solution gradually turns brown, indicating the formation of the hexamine cobalt(III) complex. After complete oxidation, cool the mixture and add concentrated hydrochloric acid (HCl) dropwise to precipitate hexamine

cobalt(III) chloride as an orange-yellow crystalline solid. The precipitate is collected using vacuum filtration, washed with cold ethanol, and dried at room temperature.

Formation of Hexammine Cobalt(II) Complex:

$$CoCl_2 + 6NH_3 \rightarrow [Co(NH_3)_6]Cl_2$$

Oxidation to Hexammine Cobalt(III) Complex:

$$2[Co(NH_3)_6]Cl_2 + H_2O_2 + 2HCl \rightarrow 2[Co(NH_3)_6]Cl_3 + 2H_2O$$

Results:

Hexamine cobalt (III) chloride was successfully synthesized as an orange-yellow crystalline solid. The expected yield was 80–90%, and the product was confirmed by its characteristic color and solubility in water.

iv. Mohr's Salt

Mohr's salt, also known as ammonium iron (II) sulfate hexahydrate, is a double salt of iron (II) sulfate ($FeSO_4$) and ammonium sulfate $(NH_4)_2SO_4$. It is commonly used as a primary standard in redox titrations due to its stability compared to $FeSO_4$, which oxidizes easily.

Procedure:

In a 250 mL beaker, dissolve 7 g of ferrous sulfate ($FeSO_4 \cdot 7H_2O$) and 3.5 g of ammonium sulfate ($(NH_4)_2SO_4$) in 50 mL of distilled water. To prevent oxidation, add a few drops of dilute sulfuric acid (H_2SO_4) to maintain an acidic medium. Heat the solution gently to 50–60°C while stirring until all the salts dissolve completely.

Allow the solution to cool slowly at room temperature and then place it in an ice bath to promote crystallization. Light green crystals of Mohr's salt start to form. The crystals are collected by vacuum filtration, washed with cold distilled water, and dried at room temperature.

$$FeSO_4 \cdot 7H_2O + (NH_4)_2SO_4 \rightarrow (NH_4)_2Fe(SO_4)_2 \cdot 6H_2O$$

Results:

Mohr's salt was successfully synthesized as pale green crystals. The expected yield was approximately 80–90%, and the product was confirmed by its characteristic color, solubility in water, and acidic nature.

v. Hexathiourea lead (II) nitrate

Hexathiourea lead (II) nitrate is a coordination complex formed by the reaction of lead (II) nitrate ($Pb(NO_3)_2$) with thiourea ($CS(NH_2)_2$) in aqueous solution. It is soluble in water and forms colourless to white crystalline precipitates.

Procedure:

In a 250 mL beaker, dissolve 3 g of lead (II) nitrate ($Pb(NO_3)_2$) in 20 mL of distilled water with gentle stirring. In another beaker, dissolve 5 g of thiourea ($CS(NH_2)_2$) in 20 mL of distilled water. Slowly add the thiourea solution to the lead (II) nitrate solution while stirring continuously. The solution is then gently heated to 50–60°C to ensure complete reaction.

After a few minutes, colourless hexathiourea lead(II) nitrate crystals begin to form. The reaction mixture is then cooled to room temperature and left undisturbed for 30 minutes to allow further crystallization. The crystals are collected by vacuum filtration, washed with **cold** distilled water, and dried in air.

$$Pb(NO_3)_2 + 6CS(NH_2)_2 \rightarrow [Pb(CS(NH_2)_2)_6](NO_3)_2$$

Results:

Hexathiourea lead (II) nitrate was successfully synthesized as colourless to white crystalline solids. The expected yield was approximately 85–90%, and the product was confirmed by its solubility in water and crystalline nature.

vi. Sodium ferrioxalate

Sodium ferrioxalate is a complex salt formed by the reaction of ferric chloride ($FeCl_3$) or ferric sulfate ($Fe_2(SO_4)_3$) with oxalic acid ($H_2C_2O_4$) and sodium oxalate ($Na_2C_2O_4$) in an aqueous medium. It is light green in color and is light-sensitive, decomposing under UV light.

Procedure:

In a 250 mL beaker, dissolve 4 g of ferric chloride ($FeCl_3 \cdot 6H_2O$) in 20 mL of distilled water with constant stirring. In another beaker, dissolve 6 g of oxalic acid ($H_2C_2O_4 \cdot 2H_2O$) in 20 mL of distilled water and heat the solution to 50–60°C. Slowly add the ferric chloride solution dropwise to the oxalic acid solution while stirring continuously. A yellow precipitate of ferric oxalate ($Fe_2(C_2O_4)_3$) forms initially.

Next, dissolve 6 g of sodium oxalate ($Na_2C_2O_4$) in 20 mL of distilled water and add it gradually to the reaction mixture. The yellow precipitate dissolves, forming a clear light green solution of sodium ferrioxalate. The solution is then cooled in an ice bath, allowing green crystals to form upon standing. The crystals are collected using vacuum filtration, washed with cold ethanol, and dried in the dark to prevent decomposition.

1. **Formation of Ferric Oxalate:**

$$2FeCl_3 + 3H_2C_2O_4 \rightarrow Fe_2(C_2O_4)_3 + 6HCl$$

2. **Formation of Sodium Ferrioxalate Complex:**

$$Fe_2(C_2O_4)_3 + 6Na^+ \rightarrow 2Na_3[Fe(C_2O_4)_3]$$

Results:

Sodium ferrioxalate was successfully synthesized as light green crystalline solid. The expected yield was 80–90%, and the product was confirmed by its solubility in water and photosensitive nature.

vii. Tristhiourea copper (I) chloride

Tris(thiourea) copper(I) chloride is a coordination complex formed by the reaction of copper(I) chloride (CuCl) with thiourea ($CS(NH_2)_2$) in aqueous solution. It appears as a white or pale yellow crystalline solid and is sensitive to air and light.

Procedure:

In a 250 mL beaker, dissolve 1 g of copper (II) chloride dihydrate ($CuCl_2 \cdot 2H_2O$) in 20 mL of distilled water with constant stirring. To reduce copper(II) to copper(I), add 2 g of sodium sulfite (Na_2SO_3) or ascorbic acid slowly while stirring. This results in the formation of white precipitate of copper(I) chloride (CuCl). The precipitate is collected by vacuum filtration, washed with cold distilled water, and kept moist to prevent oxidation.

In another beaker, dissolve 3 g of thiourea ($CS(NH_2)_2$) in 20 mL of distilled water and heat to 50°C. Add the freshly prepared CuCl to this solution while stirring continuously. The mixture is allowed to react for 15–20 minutes, forming a pale yellow or white crystalline solid. The crystals are collected using vacuum filtration, washed with cold ethanol, and dried in air away from light.

1. **Reduction of Cu(II) to Cu(I):**

$$2CuCl_2 + Na_2SO_3 + H_2O \rightarrow 2CuCl + Na_2SO_4 + 2HCl$$

2. **Formation of Tris(thiourea) Copper(I) Chloride Complex:**

$$CuCl + 3CS(NH_2)_2 \rightarrow [Cu(NH_2CSNH_2)_3]Cl$$

Results:

Tris(thiourea) copper(I) chloride was successfully synthesized as a pale yellow crystalline solid. The expected yield was approximately 80–90%, and the product was confirmed by its characteristic solubility in water and decomposition upon exposure to air and light.

viii. Sodium cobalt nitrate

Sodium cobaltinitrite is a coordination complex formed by the reaction of cobalt(III) salts with sodium nitrite ($NaNO_2$) in an acidic medium. It is a yellow crystalline solid and is commonly used as a reagent for potassium detection and as a pigment.

Procedure:

In a 250 mL beaker, dissolve 2.5 g of cobalt (II) chloride hexahydrate ($CoCl_2 \cdot 6H_2O$) or cobalt (II) nitrate hexahydrate ($Co(NO_3)_2 \cdot 6H_2O$) in 20 mL of distilled water with constant stirring. To oxidize cobalt (II) to cobalt (III), add 5 mL of 3% hydrogen peroxide (H_2O_2) dropwise while maintaining the solution at 50–60°C.

After complete oxidation, add 6 g of sodium nitrite ($NaNO_2$) dissolved in 20 mL of distilled water, followed by the slow addition of 5 mL of glacial acetic acid (CH_3COOH) to maintain an acidic medium. The solution turns yellow, indicating the formation of sodium cobaltinitrite.

Cool the reaction mixture in an ice bath, and yellow sodium cobaltinitrite crystals begin to precipitate. The precipitate is collected by vacuum filtration, washed with cold ethanol, and dried at room temperature.

1. **Oxidation of Co(II) to Co(III):**

$$2Co^{2+} + H_2O_2 + 2H^+ \rightarrow 2Co^{3+} + 2H_2O$$

2. **Formation of Sodium Cobaltinitrite Complex:**

$$Co^{3+} + 6NO_2^- + 3Na^+ \rightarrow Na_3[Co(NO_2)_6]$$

Results:

Sodium cobaltinitrite was successfully synthesized as a yellow crystalline solid. The expected yield was approximately 80–90%, and the product was confirmed by its characteristic color, solubility in water, and stability under normal conditions.

UNIT III

Determination of boiling point and melting point of organic substance / solvents.

1. Steam distillation-Extraction of essential oil from citrus fruits/eucalyptus leaves.

Aim:

To extract essential oil from citrus fruit peels (such as orange or lemon) or eucalyptus leaves using steam distillation and to study the process of separating volatile compounds from plant materials.

Procedure:

Fresh citrus peels or eucalyptus leaves were collected and finely chopped to increase the surface area for efficient oil extraction. A round-bottom flask was filled with distilled water, and the prepared plant material was added. The flask was then connected to a condenser and a receiver flask to collect the distillate. In some cases, a steam generator was used to introduce steam directly into the flask. The mixture was gently heated until steam formed, carrying the volatile essential oil components along with it. As the steam-oil mixture passed through the condenser, it cooled and condensed into a liquid, which was collected in the receiver flask as a mixture of water and essential oil.

To separate the essential oil, the distillate was transferred into a separatory funnel and left undisturbed to allow phase separation. Since essential oils are immiscible with water, they formed a distinct layer, which was carefully extracted. To remove any residual moisture, the oil was dried using anhydrous sodium sulfate. Finally, the extracted essential oil was stored in a clean, dry, airtight glass vial and kept in a cool, dark place to prevent degradation and preserve its aromatic properties.

Result:

The essential oil was successfully extracted from citrus fruit peels or eucalyptus leaves using steam distillation. The extracted oil appeared pale yellow (for citrus) or colourless to pale green (for eucalyptus) with a strong characteristic aroma. The typical yield of essential oil was observed to be **0.5–3%**, depending on the plant material used.

2. Chromatography (anyone (Group experiment)

(i) Separation of amino acids by Paper Chromatography

(ii) Thin Layer Chromatography-mixture of sugars/plant pigments/permanganate, dichromate.

(iii) Column Chromatography-extraction of carotene, chlorophyll and xanthophylls from leaves /separation of anthracene-anthracene picrate.

(i) Separation of Amino Acids by Paper Chromatography

Aim:

To separate and identify different amino acids in a mixture using paper chromatography.

Procedure:

A strip of Whatman No. 1 filter paper was taken, and a pencil line was drawn 2 cm above the bottom to mark the baseline. Small spots of an amino acid mixture and reference amino acids were applied on the line using a capillary tube and allowed to dry. The paper was placed in a chromatography chamber containing a solvent system (e.g., n-butanol, acetic acid, and water in a 4:1:5 ratio**)** with the baseline above the solvent level. As the solvent moved up by capillary action, the amino acids migrated at different rates based on their solubility and interaction with the paper. After the solvent reached the top, the paper was removed, dried, and sprayed with ninhydrin reagent**,** which developed colored spots. The Rf values were calculated and compared with standard amino acid values.

Result:

Different amino acids were successfully separated and identified based on their Rf values and color reactions.

(ii) Thin Layer Chromatography (TLC) – Separation of Sugars / Plant Pigments / Permanganate & Dichromate

Aim:

To separate components of a mixture (e.g., sugars, plant pigments, permanganate, or dichromate) using thin-layer chromatography (TLC).

Procedure:

A TLC plate (coated with silica gel as the stationary phase) was used. A pencil

baseline was marked 2 cm from the bottom, and a sample solution (e.g., plant extract, sugar solution, or inorganic ions) was applied as a small spot. The plate was placed upright in a chromatography chamber containing an appropriate mobile phase (e.g., ethyl acetate: hexane for pigments or a suitable solvent for sugars). As the solvent moved up the plate via capillary action, different components travelled at varying rates, leading to separation. Once the solvent front reached the top, the plate was removed and dried. The separated spots were visualized using UV light, iodine vapours, or a suitable staining reagent (e.g., ninhydrin for amino acids, ferric chloride for sugars).

Result:

Different components of the mixture were effectively separated as distinct spots on the TLC plate, identified based on their Rf values and appearance.

(iii) Column Chromatography – Extraction of Carotene, Chlorophyll, and Xanthophylls from Leaves

Aim:

To separate plant pigments (carotene, chlorophyll, and xanthophylls) from leaf extracts using column chromatography.

Procedure:

Fresh green leaves were crushed and extracted with acetone to obtain a pigment solution. A chromatography column was packed with silica gel as the stationary phase, and the leaf extract was carefully loaded onto the column. A suitable mobile phase (e.g., a mixture of petroleum ether and acetone) was added slowly, allowing the pigments to separate based on their polarity. Carotene (yellow-orange), chlorophyll (green), and xanthophylls (yellow) were observed as distinct bands moving through the column at different rates. The fractions were collected separately for further analysis.

Result:

The pigments were successfully extracted and separated based on their polarity, with carotene eluting first, followed by xanthophylls and chlorophyll.

3. Electrophoresis–Separation of amino acids and proteins. Isolationofcaseinfrommilk/Determinationofsaponificationvalueofoilor fat/Estimation of acetic acid from commercial vinegar.

Electrophoresis – Separation of Amino Acids and Proteins

AIM:

To separate amino acids and proteins based on their charge and molecular weight using electrophoresis.

PROCEDURE:

A buffer solution of appropriate pH is prepared and poured into the electrophoresis chamber. A sample mixture of amino acids or proteins is loaded onto the gel or paper strip. The electrodes are connected, and a voltage is applied across the system. The charged molecules migrate toward the electrode of opposite charge based on their isoelectric points and size. After the required time, the movement of different amino acids or proteins is analysed under UV light or stained for better visibility.

RESULTS:

Different amino acids and proteins migrated at varying distances, confirming their separation based on charge and size. The negatively charged molecules moved towards the anode, while positively charged ones moved towards the cathode.

Isolation of Casein from Milk

AIM:

To isolate casein protein from milk using an acid precipitation method.

PROCEDURE:

A measured volume of milk is heated to about 40°C, and dilute acetic acid (or lactic acid) is added dropwise while stirring continuously. The casein precipitates as a white solid. The mixture is filtered to separate casein, which is then washed with water and dried.

RESULTS:

A white precipitate of casein was successfully isolated from milk.

Determination of Saponification Value of Oil or Fat

AIM:

To determine the saponification value of a given fat or oil by titration.

PROCEDURE:

A known mass of the oil or fat sample is refluxed with an excess amount of alcoholic KOH solution. The unreacted KOH is then titrated against standard hydrochloric acid (HCl) using phenolphthalein as an indicator. The saponification value is calculated based on the volume of KOH consumed.

RESULTS:

The saponification value was determined for different oil samples, indicating their fatty acid composition. Higher values suggest smaller molecular weight fatty acids.

Estimation of Acetic Acid from Commercial Vinegar

AIM:

To determine the percentage of acetic acid in commercial vinegar using acid-base titration.

PROCEDURE:

A measured volume of vinegar is titrated against a standard sodium hydroxide (NaOH) solution using phenolphthalein as an indicator. The amount of NaOH required to neutralize the acetic acid is recorded. The percentage of acetic acid is calculated using the titration formula.

RESULTS:

The percentage of acetic acid in commercial vinegar was determined and compared across different samples.