PRACTICAL LAB MANUAL

for

B PHARM

II SEM

Subject:

PHARMACEUTICAL ORGANIC CHEMISTRY

BP 208P

Name :

Roll No. :

Class :

Course :

Semester :

Academic Year :

Experiment - 1

Object – To determine the melting point of given organic compound.

References :

1. Vogel Arthere, Elementary practical organic chemistry, 2nd edition, 1966, published by CBS publisher, page No. 3-12.
2. Agarwal O.P., Practical Organic chemistry, published by Krishna educational publisher, Edition 2014, page no. 60-64.

Requirements – Given Solid Substance, Capillary Tube, Melting Point apparatus.

Theory –

The change from solid to liquid state of a compound in heating is called melting and the temperature at which a solid in its pure form melts is called the melting point. Every pure solid has a characteristics melting point therefore determination of melting point helps in identification of the compound.

Presence of impurities lowers the melting point of the solid. Thus Melting point also serves as a criterion of purity of a compound.

Procedure –

1. Take a fine capillary of length 5-6cm.
2. Seal its one end by inserting the end of the capillary tube horizontally into the extreme edge of a small steady Bunsen flame for a few seconds, rotating the capillary mean while.
3. Take a small quantity of the compound whose melting point is to be determined in mortar pestle and powder it.
4. Introduce the powdered compound in the capillary tube.
5. Gently tap the capillary tube so that the compound sinks into the closed end.
6. Repeat the procedure of introducing and tapping three to four times.
7. Put the capillary in digital melting point apparatus.
8. observe the temp. at which it start to melt.
9. Finally note the temp. at which it completely melt.

Observations –

	Melting point
1	..... °C
2	..... °C
3	..... °C

$$\text{Mean Melting point} = \frac{(t_1 + t_2 + t_3)}{3} \ ^\circ C$$

Result : The melting point of the given organic compound is .....

Precaution –

1. The capillary tube is filled one third of its length.
2. The rate of heating should be controlled.
3. It should be very slow near the melting point so that the melting point can be recorded accurately.

Melting point of some common organic compounds

• Phenol	: 42 °C	• Benzoic Acid	: 122 °C
• α-Naphthylamine	: 50 °C	• Fructose	: 103 °C
• α-Naphthol	: 96 °C	• Cinnamic Acid	: 133 °C
• Acetamide	: 82 °C	• Glucose	: 146 °C
• β-Naphthol	: 123 °C	• p-Toludine	: 43 °C
• Benzamide	: 128 °C	• Sucrose	: 186 °C
• Oxalic Acid	: 101 °C	• Naththalene	: 80 °C
• Urea	: 132 °C	• Acetanilide	: 114.3 °C

EXPERIMENT 02

Object – Qualitative analysis of unknown organic compound/s for preliminary tests.

Reference –

1. Vogel Arthere, Elementary practical organic chemistry, 2nd edition, 1966, published by CBS publisher, page No. 61-64.
2. Agarwal O.P., Practical Organic chemistry, published by Krishna educational publisher, Edition 2014, page no. 31-32.

Requirement –

Test tube, spatula, different compound, glass-rod etc

Theory –

Preliminary qualitative analysis of an unknown organic compound involves initial tests to determine the compound's general properties and the presence of specific elements and functional groups. These tests include physical examination, solubility tests, and flame tests.

	Test	Observation	Inferences
1.	(a) Nature	i) Solid	Carbohydrate, acid, phenol, amine, higher hydrocarbon may be present.
		ii) Liquid	Alcohol, ketone, aldehyde, ester, phenol, amines may be present.
2.	(b) Colour	Yellow - Solid	m - Dinitrobenzene, p - Nitro toluene, nitro phenol, nitro aniline
		Yellow - liquid	Nitrobenzene.
		Brown	P - Toluidine, resorcinol.
		Blackish	α - Naphthol
		Pink	β - Naphthol
		Buff or reddish	Aniline, phenol, Aromatic amine.
		Colourless	Simple acid, alcohol, ester, ketone aromatic hydrocarbon.
3.	Odour	Carbolic	Phenol, cresol.
		Fishy	Amine
		Sweet pleasant	Ester, alcohol and halogen derivatives.
		Bitter almonds	Nitrobenzene, Benzaldehyde
		Moth balls	Naphthalene
		No particular smell	Aromatic acid, amide, carbohydrate
4.	Flame Test	Sooty flame	Aromatic compound or aliphatic compound containing small proportion of hydrogen e.g. $CHCl_3$, CCl_4
		Non sooty flame	Aliphatic compound
		Substance chars	Carbohydrate, sulphanilic acid.
5.	Test for unsaturation	1. $KMnO_4$ test. Substance + 2 ml of water shake well + 2 drops of dilute $KMnO_4$ solution	
		Decolourisation of $KMnO_4$	Saturated compound.
		No decolourisation	Unsaturated compound.

Result : Qualitative analysis of unknown organic compound/s for preliminary tests was done successfully.

EXPERIMENT 03

Object – Qualitative analysis of unknown organic compound/s by Lassaigne tests (Nitrogen).

Reference –

1. Vogel Arthere, Elementary practical organic chemistry, 2nd edition, 1966, published by CBS publisher, page No. 35
2. Agarwal O.P., Practical Organic chemistry, published by Krishna educational publisher, Edition 2014, page no. 24-27.

Requirement – Ignition tube, capillary tube, pipette, test tube, china dish, filter paper, funnel, unknown organic compound (urea)

Theory –

The Lassaigne's test, also known as the sodium fusion test, is a qualitative analysis technique used to detect the presence of nitrogen, sulfur, and halogens in an organic compound.

It utilizes the reactivity of sodium with the elements nitrogen, sulfur, and halogens in organic compounds. When heated with sodium, these elements form water-soluble salts, which are then extracted and tested using specific chemical reactions.

Procedure –

Sodium Fusion Test (Lassaigne's Test)

1. Take a small piece of dry sodium metal in a fusion tube and heat it gently till the metal melts or fuses.
2. Add equal quantity of compound to this fused metal [If the compound is a liquid then add two drops of it with a capillary]
3. Heat it gently then strongly till it becomes red hot.
4. Plung the red hot tube in 10 ml or ¾ of a test tube of distilled water taken in a porcelain dish, covering it immediately with an asbestos sheet crush the fusion tube completely.
5. Carry out more fusion in the similar way. Boil the extract for five minutes reduce the volume to about 5 ml and filter. Perform following test using this filtrate.
6. 1 ml of extract + 2-3 drops of NaOH solution to make it alkaline.
7. Add a few drops of freshly prepared $FeSO_4$ solution, boil for a few minutes, cool and acidify it with by adding dil. HCl or dil. H_2SO_4.
8. Blue or green colour solution or Prussian blue coloration.

Inference –

If Prussian blue color is present ⟶ Nitrogen is present

Result :

Qualitative analysis of unknown organic compound/s by lassaign tests (Nitrogen) was done successfully.

EXPERIMENT 04

Object : Qualitative analysis of unknown organic compound/s by Lassaigne tests (Sulphur).

Reference :

1. Agarwal O.P., Practical Organic chemistry, published by Krishna educational publisher, Edition 2014, page no. 26.
2. Dr. Peesapati V., A comprehensive approach to practical organic chemistry, published by PharmaMed Press, 2023, pg No 35.

Requirement : Ignition tube, capillary tube, pipette, test tube, china dish, filter paper, funnel, unknown organic compound (sod. nitro prusside, lead acetate).

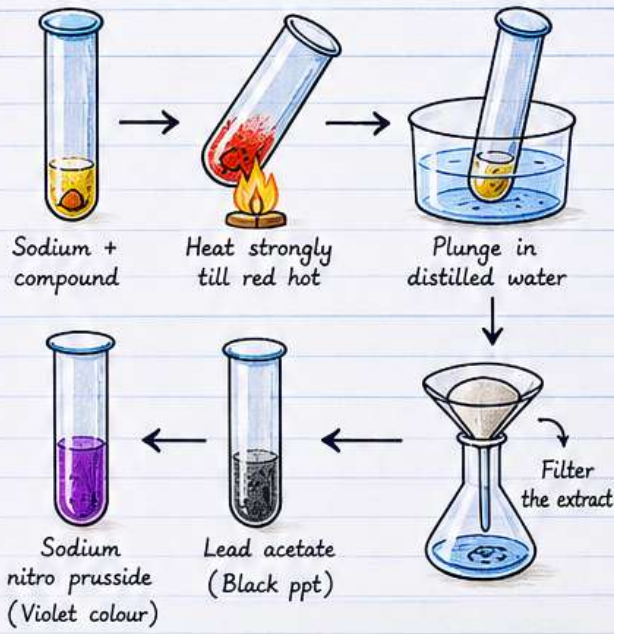
Theory : The Lassaigne's test, also known as the sodium fusion test, is a qualitative analysis technique used to detect the presence of nitrogen, sulfur, and halogens in an organic compound.

It utilizes the reactivity of sodium with these elements. When heated with sodium, these elements form water-soluble salts, which are then extracted and tested using specific chemical reactions.

Procedure :

1. Take a small piece of dry sodium metal in a fusion tube and heat it gently till the metal melts.
2. Add equal quantity of unknown compound to this fused sodium.
3. Heat it gently and then strongly till the tube becomes red hot.
4. Plunge the red hot tube in 10 ml distilled water taken in a china dish, covering it immediately with an asbestos sheet and crush the fusion tube completely.
5. Boil the extract for 5 minutes, reduce the volume to about 5 ml and filter.
6. To 2-3 ml of filtrate add 2-3 drops of lead acetate solution.
7. Formation of black precipitate indicates the presence of Sulphur.
8. Confirm by sodium nitro prusside test.
   A few drops of sodium nitro prusside solution will produce violet colour with black ppt.

Lassaigne's Test for Sulphur



Observations :

Test	Observation	Inference
1. Lead acetate test	Black precipitate formed	Sulphur is present
2. Sodium nitro prusside test	Violet colour obtained	Sulphur is confirmed

Chemical Reaction :

$S^{2-} + Pb(CH_3COO)_2 \rightarrow PbS \downarrow$ (Black ppt.)

$Fe^{2+} + [Fe(CN)_5NOS]^{2-} \rightarrow$ Violet complex

Inference : If black precipitate with lead acetate and violet colour with sodium nitro prusside are obtained → Sulphur is present.

Result : Qualitative analysis of unknown organic compound/s by Lassaigne tests (Sulphur) was done successfully.

EXPERIMENT No. 5

Carboxylic acid

Object : Qualitative Analysis of unknown organic compound for function group detection (Carboxylic acid).

Reference :
1. Agarwal O.P., Practical Organic chemistry, published by Krishna educational publisher, Edition 2014, page no. 30-31
2. Dr. Jain K.S., Dr. Miniyar P.B., A practical book of Pharmaceutical organic chemistry, Nirali publication, edition 4th, page No. 1.21-1.23

Requirement : Test tube, Beaker, Glass rod
Organic compound, Sod bicarbonate, HCl

Theory : Organic compounds containing carboxyl functional groups are called carboxylic acids. The term carboxyl, derives its name from the combination of words carbonyl and hydroxyl because carboxylic functional group O contains both of these groups ($-\overset{O}{\overset{\|}{C}}-OH$). These acids turn blue litmus red and react with sodium hydrogencarbonate solution to produce effervescence due to the formation of carbon dioxide. This is a test that distinguishes carboxylic acids from phenols.

Carboxyl Group

$R-\overset{O}{\overset{\|}{C}}-OH$

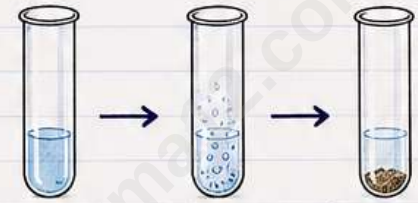
(-COOH)

Procedure :

1. SODIUM BICARBONATE TEST

- Take 0.2 g / 0.2 ml of unknown organic compound in test tube.
- Add 2-3 ml of aqueous $NaHCO_3$ (10%).

Reaction : $R-COOH + NaHCO_3 \longrightarrow R-COONa + CO_2\uparrow + H_2O$



Compound + $NaHCO_3$ → Effervescence (CO_2) → After adding dil. HCl solid reappears

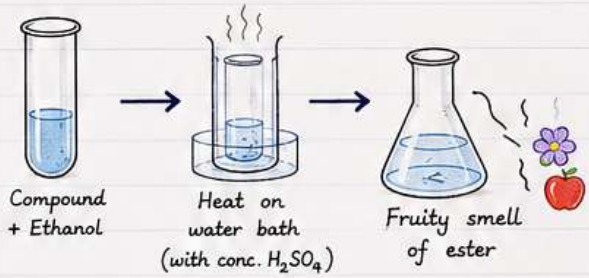
Observation :
1. Effervescence of CO_2
2. Reappearance of the solid substance after adding dil HCl.

Inference : COOH group is present.

2. ESTER TEST

- Take 0.2 gm/0.2 ml of compound in test tube.
- Add 5 ml of ethanol.
- Add 2 drops of con. H_2SO_4.
- Heat it on boiling water bath.

Reaction : $R-COOH + C_2H_5OH \xrightarrow{H_2SO_4} \longrightarrow R-COOC_2H_5 + H_2O$



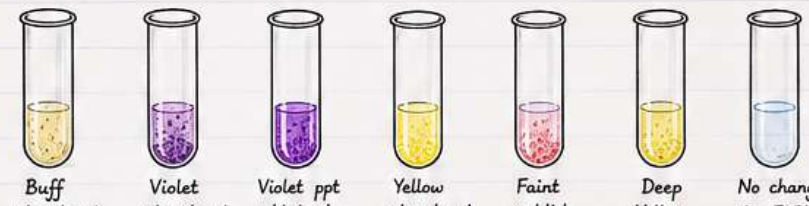
Compound + Ethanol → Heat on water bath (with conc. H_2SO_4) → Fruity smell of ester

Observation : Fruity smell of ester.

Inference : -COOH group is present.

3. FERRIC CHLORIDE TEST

- Take 0.05 gm of compound.
- Add 1 ml of water and shake well.
- Add 1-2 drops of alcoholic $FeCl_3$ solution.

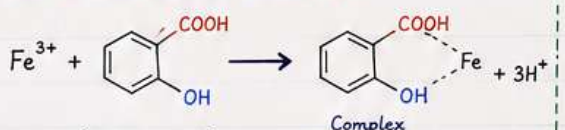


Buff colored ppt	Violet colored ppt	Violet ppt obtained after heating	Yellow colored ppt	Faint raddish	Deep Yellow	No change in $FeCl_3$
Benzoic acid/ pthalic acid	Salicylic acid	Acetyl Salicylic acid	Cinnamic acid	Succinic acid	Citric acid	Oxallic acid

Observation :

Observation	Inference
Buff colored precipitate	Benzoic acid/pthalic acid
Violet colored precipitate	Salicylic acid
Violet colored ppt obtained after heating	Acetyl Salicylic acid
Yellow colored ppt	Cinnamic acid
Faint raddish	Succinic acid
Deep Yellow	Citric acid
No change in $FeCl_3$	Oxallic acid

Ferric chloride test is based on :



Fe^{3+} + (Salicylic acid) → Complex (Violet colour) + $3H^+$

Result : _____ functional group was found in given organic compound.

Experiment No. 6

Objective

Qualitative Analysis of unknown organic compound for detection of carbohydrate

References:

1. Dr. Jain K.S., Dr. Miniyar P.B., A practical book of Pharmaceutical organic chemistry, Nirali publication, edition 4th, page No. 1.31-1.33
2. Vogel Arthere, Elementary practical organic chemistry, 2nd edition, 1966, published by CBS publisher, page No. 89

Requirement

A. Glasswares and Instrument

1. Test tubes
2. Pipette or dropper
3. Beaker (for water bath)
4. Test tube holder
5. Bunsen burner or hot water bath

B. Chemicals

1. Molisch Reagent (α-naphthol in ethanol)
2. Concentrated sulfuric acid (H_2SO_4)
3. Fehlind Sol A (Copper(II) sulfate solution) & B (Alkaline solution of potassium sodium tartrate (Rochelle salt) in sodium hydroxide)
4. Benedict Reagent ((contains copper(II) sulfate, sodium carbonate, and sodium citrate))
5. Sample (glucose, fructose, sucrose)
6. Distilled water

Theory:

Carbohydrates are organic compounds containing carbon, hydrogen, and oxygen. They include simple sugars (monosaccharides), disaccharides, and polysaccharides. Molisch's test is commonly used as a general test. This test is based on the dehydration of carbohydrates by concentrated sulfuric acid to form furfural or hydroxymethylfurfural, which then react with α-naphthol (Molisch's reagent) to produce a purple or violet ring at the interface of the acid and the aqueous layer.

In Fehling's Test: Reducing sugars react with the copper ions in Fehling's reagent to form copper(I) oxide (Cu_2O), which precipitates out as a brick-red solid which indicates the presence of reducing sugars.

In Benedict's Test: Similar to Fehling's test, reducing sugars reduce the copper(II) ions in Benedict's reagent to copper(I) oxide, which precipitates out. The reagent changes color, ranging from green, yellow, orange, or red, depending on the

Procedure

Molisch Test

1. Take 2 mL of the aqueous carbohydrate solution in a clean test tube.
2. Add 2-3 ml of Molisch's reagent (α-naphthol in ethanol) to the solution.
3. Carefully add 1-2 drops of concentrated sulfuric acid along the sides of the test tube using a dropper or pipette. Do not mix. Allow the acid to settle at the bottom.
4. Observe the formation of a violet or purple ring at the junction of the two liquids.

D-glucose + con H_2SO_4 ⟶ 5 Hydroxy methyl furfural ⟶
+ alpha nephthol ⟶ purple/violet ring

Fehling's Test:

1. Mix 1 mL of Fehling's solution A and 1 mL of Fehling's solution B in a test tube to prepare fresh Fehling's reagent.
2. Add 2 mL of the carbohydrate solution (or a few crystals of the solid sample dissolved in water) to the test tube.
3. Gently heat the mixture in a boiling water bath for 2-5 minutes or directly over a low flame with a test tube holder.
4. Observe any color change or precipitate (red) formation.

Benedict Test

1. Take 2 mL of the carbohydrate solution in a clean test tube.
2. Add 2 mL of Benedict's reagent to the test tube.
3. Mix well and heat the solution in a boiling water bath for 3-5 minutes, or gently over a flame.
4. Observe the color change or precipitate formation.
5. The degree of color change can give a rough estimate of sugar concentration:
   - Blue (no change): No reducing sugar
   - Green: Trace amount
   - Yellow: Low concentration
   - Orange: Moderate concentration
   - Brick-red: High concentration

Observation

Molisch Test: ..... color is found
Fehling Test: ..... color is found
Benedict Test: ..... color is found

Result: Qualitative analysis of organic compound for detection of carbohydrate was done successfully.

EXPERIMENT 07

Object

To prepare Iodoform from acetone

Reference

1. Vogel Arthere, Elementary practical organic chemistry, 2nd edition, 1966, published by CBS publisher, page No. 198
2. Bahl A & Bahl B S, A text book of Organic chemistry, published by s chand and company limited, Edition: 22, page No. 309

Requirement

Conical flask, beaker, measuring cylinder

chemical:

Acetone	5ml
Iodine	5 gram
NaOH solution	5% w/v

Theory

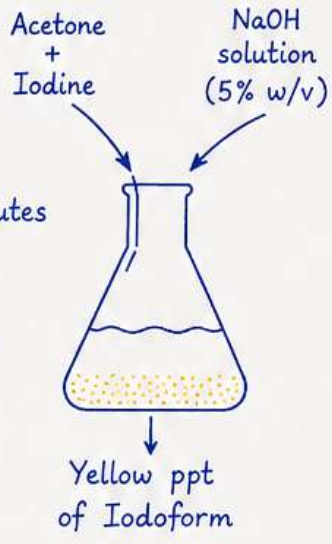
Iodoform, with the chemical formula CHI_3, is a pale yellow, crystalline, volatile substance known for its distinctive, sweetish odor. It is an organoiodine compound and was historically used as a disinfectant and antiseptic in medicine.

Iodoform also known as tri-iodo-methane is prepared from acetone and iodine crystals in the presence of aqueous sodium hydroxide solution

$$CH_3COCH_3 + 4I_2 + 4NaOH \longrightarrow \underset{\text{(Iodoform)}}{CHI_3 \downarrow} + 3NaI + CH_3COONa + 2H_2O$$

Procedure

1. Dissolve 5g of iodine in 5ml of acetone in a conical flask
2. Add 5% sodium hydroxide solution slowly with continuous shaking until all the iodine color is discharged
3. Allow the content of the flask to stand for about 10-15 minutes
4. Filter off the yellow crystalline ppt of Iodoform though Buchner funnel
5. Wash with little water and dried between the filter paper and weighed



Observation:

Yellow color precipitate obtained.

Result:

Iodoform prepared successfully

EXPERIMENT 08

Object

To prepare Phenyl benzoate from phenol

Reference

Peesapati venkateshwarlu, Elementary practical organic chemistry, 2nd edition, 1966, published by CBS publisher, page No. 172

Requirement

Iodine flask, beaker, measuring cylinder, pipette

chemical:

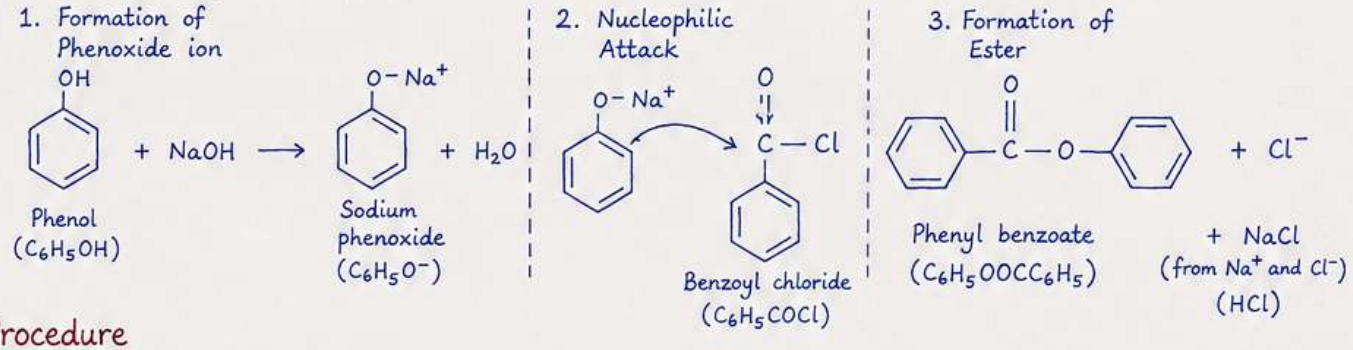
Benzoyl chloride	2 ml
Phenol	1gram/ 1ml
NaOH solution	10% w/v
ethyle alcohol	

Theory.

Phenyl benzoate is synthesized by reacting phenol with benzoyl chloride in the presence of a base like sodium hydroxide, utilizing the Schotten-Baumann reaction. This method leverages the nucleophilic nature of the phenoxide ion, formed when phenol reacts with the base, to attack the electrophilic carbonyl carbon of benzoyl chloride, resulting in the formation of the phenyl benzoate ester.

1. Formation of Phenoxide Ion:
   Phenol (C_6H_5OH). reacts with sodium hydroxide (NaOH) to form a phenoxide ion ($C_6H_5O^-$) and water. This reaction is facilitated by the strong base, which deprotonates the phenol, making the oxygen atom a better nucleophile.
2. Nucleophilic Attack:
   The phenoxide ion, with its negative charge, acts as a nucleophile and attacks the electrophilic carbonyl carbon of benzoyl chloride (C_6H_5COCl).
3. Formation of the Ester:
   This attack leads to the displacement of the chloride ion (Cl^-) from benzoyl chloride, forming phenyl benzoate ($C_6H_5OOCC_6H_5$) and hydrochloric acid (HCl).
4. Schotten-Baumann Method:
   The reaction is typically carried out under basic conditions (using sodium hydroxide) and with vigorous shaking to ensure good mixing and reaction progress.

The phenyl benzoate, being insoluble in water, precipitates out as a solid, allowing for easy separation from the aqueous solution.



Procedure

1. 1 gram of phenol is dissolved in 15 mL of 10% sodium hydroxide solution in an Erlenmeyer flask.
2. 2 mL of benzoyl chloride is added.
3. Close the mouth of the flask with a rubber stopper and shake vigorously for about 30 minutes.
4. filter the crystal and recrystalize it with ethyle alcohol.

Result

Phenyl benzoate prepared from phenol.

EXPERIMENT 09

Object :

To Prepare picric acid from phenol

Reference :

1. Agarwal O.P., Practical Organic chemistry, published by Krishna educational publisher, Edition 2014, page no. 316
2. Mann F.G., Saunders B.C., "practical organic chemistry, published by orient hangmen private ltd., 4th edition, page N. 173

Requirement :

Glassware : Conical Flask, Glass rod, Measuring Cylinder, Pipette

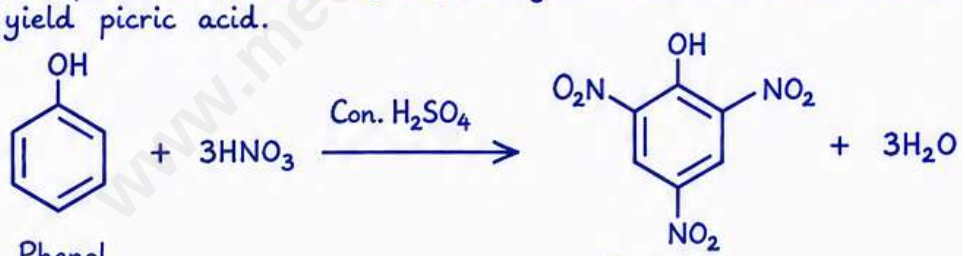
Chemicals : Phenol, Con. H_2SO_4, Con. HNO_3

Theory :

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.

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 electrophillic aromatic substitution.

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 electrophillic aromatic substitution, the nitration reaction occurs very easily. It undergoes nitration with HNO_3 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.



Phenol

Picric acid
(2,4,6-Trinitro phenol)

Procedure :

1. Place 4 gm of phenol to conical flask and add 5 ml of Con. H_2SO_4.
2. Mix the solution properly (solution become warm).
3. Heat the flask on water bath for 30 minutes.
4. After 30 minutes place the flask on Ice bath.
5. Now add 20 ml of Con. HNO_3 & shake it properly.
6. Mixture allow to stand undisturbed until red fumes stops poring out.
7. When fumes stop, heat the flask on water bath for 1-2 hrs with occassional stirring.
8. After given time add 100 ml of cold water and chill the mixture.
9. Filter the yellow crystals.
10. Wash with water to remove excess of inorganic acids.

Observation :

Yellow colored crystal are obtained.

Result :

Picric acid was prepared successfully.

EXPERIMENT 10

OBJECT : To prepare molecular model of different compound.

REFERENCE :

1. Arora varun, Pharmaceutical organic chemistry-I, published by medical publisher, 2022, Pg.No. 387-392
2. Dr. Jain KS, Practical book of pharmaceutical chemistry, published by Nirali prakashan, 4th edition, 2020, Pg.No. 2.1-2.8

REQUIREMENT :

Model kits

THEORY :

In this experiment we will use ball and stick models to represent atoms and bonds in molecules and build molecular structures using these models.

In order to better understand chemical properties of molecules, it will help to be able to visualize the three dimensional structure.

The model kits consist of different colored balls with holes for the pegs that connect them. The balls are color coded and the holes correspond to the number of bonds each atom normally has.

We will not be using all of the different balls (atoms) provided in the kit. Notice that the number of holes in each ball corresponds to what chemists often refer to as the valence of an atom (in this case the usual valence for organic molecules).

Hydrogen and the halogens, chlorine, bromine and iodine, have only one bond to carbon. Oxygen forms two covalent bonds, nitrogen usually forms three covalent bonds, and carbon always forms 4 covalent bonds.

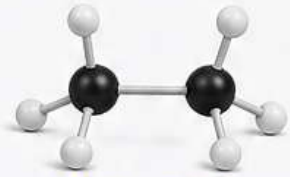
This gives rise to what is sometimes called the HONC rule; i.e., H has one bond, O has 2, N has 3 and C has 4 bonds in most neutral organic molecules.

MAKING OF MOLECULES :

1. CO_2

($O = C = O$)

2. Ethane (C_2H_6)



($H_3C - CH_3$)

3. Propane (C_3H_8)

($CH_3 - CH_2 - CH_3$)

4. Methane (CH_4)

(CH_4)

5. Butene (C_4H_8)

($CH_3 - CH = CH - CH_3$)

RESULT :

Molecular model of organic compounds were prepared successfully.

PREPARATION OF REAGENTS

Benedict's Reagent

Requirements (Chemicals):

- Copper(II) sulfate pentahydrate ($CuSO_4 \cdot 5H_2O$) – 17.3 g
- Sodium carbonate (Na_2CO_3) – 100 g (anhydrous)
- Sodium citrate ($C_6H_5Na_3O_7 \cdot 2H_2O$) – 173 g
- Distilled water – To make 1 liter

Theory Behind Components:

- Copper(II) sulfate provides the Cu^{2+} ions (blue color), which are reduced by reducing sugars.
- Sodium carbonate creates an alkaline medium, necessary for the redox reaction.
- Sodium citrate acts as a complexing agent, preventing precipitation of copper carbonate in alkaline solution by keeping Cu^{2+} in solution.

Procedure:

1. Dissolve sodium carbonate (100 g) and sodium citrate (173 g) in about 800 mL of distilled water in a beaker or volumetric flask. Stir well until fully dissolved.
2. In a separate container, dissolve copper(II) sulfate pentahydrate (17.3 g) in about 100 mL of distilled water.
3. Slowly add the copper sulfate solution to the carbonate-citrate solution with constant stirring.
4. Once completely mixed, make up the volume to 1 liter with distilled water.
5. Store the reagent in a dark bottle (amber glass) and label properly. The reagent is stable for several months at room temperature.

Storage:

- Store in a cool, dark place.
- Always shake well before use.
- Discard if any precipitate forms or color fades significantly.

PREPARATION OF REAGENTS

Molisch's Reagent

Requirements (Chemicals):

- α-Naphthol (alpha-naphthol) – 5 g
- Ethanol (95% or absolute alcohol) – 100 mL

Procedure :

1. Take a clean, dry amber glass bottle.
2. Dissolve 5 grams of α-naphthol in 100 mL of ethanol.
3. Shake well until the α-naphthol is completely dissolved.
4. Label the bottle: "Molisch's Reagent – 5% α-Naphthol in Ethanol".
5. Store the reagent in a cool, dark place, tightly stoppered.

Note:

- Use pure α-naphthol for best results.
- The reagent is flammable due to ethanol – handle with care.
- Store in an amber bottle to prevent degradation from light exposure.

Object

To synthesize aspirin (acetylsalicylic acid) from salicylic acid

Reference:

Principle

Aspirin is prepared by the acetylation of salicylic acid. The hydroxyl (-OH) group of salicylic acid reacts with acetic anhydride in the presence of a few drops of concentrated sulfuric acid or phosphoric acid. The reaction produces acetylsalicylic acid (aspirin) and acetic acid as a by-product.

Chemical Reaction

Salicylic Acid + Acetic Anhydride $\longrightarrow$ Aspirin + Acetic Acid

Requirements

Chemicals

- Salicylic acid – 2 g
- Acetic anhydride – 4–5 mL
- Concentrated sulfuric acid or phosphoric acid – 2–3 drops
- Distilled water
- Ethanol (for recrystallization)

Apparatus

- Conical flask
- Beaker
- Measuring cylinder
- Glass rod
- Water bath
- Funnel
- Filter paper
- Watch glass

Procedure

1. Weigh 2 g of salicylic acid and transfer it to a clean, dry conical flask.
2. Add 4–5 mL of acetic anhydride to the flask.
3. Add 2–3 drops of concentrated sulfuric acid (or phosphoric acid) as a catalyst.
4. Mix the contents gently by swirling the flask.
5. Heat the mixture in a water bath at 50–70°C for about 10–15 minutes.
6. Remove the flask and allow it to cool slightly.
7. Add 20–30 mL of cold distilled water slowly to destroy the excess acetic anhydride.
8. Cool the mixture in an ice bath until white crystals of aspirin are formed.
9. Filter the crystals using filter paper and wash them with a small quantity of cold water.

Observation

- White crystalline aspirin is obtained after cooling.
- The purified product has a melting point of 135–136°C.

Result

Aspirin (acetylsalicylic acid) was successfully synthesized from salicylic acid