S.A.RAJA PHARMACY COLLEGE

VADAKKANGULAM 627 116

SUBJECT: PHARMACEUTICAL ORGANIC CHEMISTRY

II SEMESTER B.PHARM

PRACTICAL LAB MANUAL

Ex.No:
Date:

PREPARATION OF BENZOIC ACID

Aim:

To prepare and submit benzoic acid from benzamide

Principle:

Benzoic acid is an aromatic acid. It can be prepared by the reaction of benzamide with sodium hydroxide which on hydrolysis gives sodium benzoate and ammonia. It is then acidified with conc. HCl to give benzoic acid.

Chemical required:

Benzamide	-	1.5g
10% NaOH	-	10ml
Conc.HCl	-	q.s

Procedure:

Take 1.5g of benzamide and 10% NaOH 10ml in a round bottom flask which are mixed well with constant stirring and heated in a water bath until ammonia gas ceases completely. Cool the beaker and add 20ml of distilled water and then add Conc.HCl drop by drop till the solution becomes acidic. Filter the solution and wash with water to remove all impurities and finally dried and weighed.

Recrystallisation:

It is recrystallised by using hot water.

Report:

The amount of benzoic acid is obtained =

Chemical reaction:

Ex.No:
Date:

PREPARATION OF IODOFORM

Aim:

To prepare and submit iodoform from acetone

Principle:

The principle involved in the preparation of iodoform is halogenation. When methyl ketone or aldehyde having CH3C=o group, ethanol, is treated with halogen in the presence of base like sodium hydroxide, it undergoes aliphatic substitution to form a salt of haloform. In this preparation acetone reacts with iodine in the presence of base like sodium hydroxide to give iodoform, sodium acetate, sodium iodide.

Chemical required:

Acetone	-	3ml
10% iodine solution	-	10ml
10% NaOH	-	q.s

Procedure:

Take 3ml of acetone and 10ml of 10% iodine solution and add 10% NaOH drop by drop with continuous stirring in a beaker until to get iodoform as a lemon yellow crystalline solid. Filter the solution and wash with cold water and finally dried and weighed.

Recrystallisation:

It is recrystallised by using a mixture of methanol and water in the ratio 1:1

Report:

The amount of iodoform is obtained =

Chemical reaction:

$CH_3\text{-}Co\text{-}CH_3 + 3I_2 + 4NaOH \rightarrow CHI_3 + CH_3COONa + 3NaI + 3H_2O$

(Acetone) (Iodine) (Iodoform) (Sodium acetate) (Sodium iodide)

Ex.No:
Date:

PREPARATION OF BENZOIC ACID

Aim:

To prepare and submit salicylic acid from methyl salicylate

Principle:

Salicylic acid is an aromatic acid. It can be prepared when methyl salicylate is heated with sodium hydroxide which on hydrolysis gives sodium salicylate and methanol. This sodium salicylate undergoes acid hydrolysis with conc. HCl to give salicylic acid.

Chemical required:

Methyl salicylate	-	2ml
10% NaOH	-	10ml
Conc.HCl	-	q.s

Procedure:

Take 2ml of methyl salicylate and10ml of 10% NaOH in a 10ml beaker or round bottom flask and mix well and heat. The mixture in a water bath until oily layer of methyl salicylate is disappears. To the mixture add Conc.HCl drop by drop until the solution becomes acidic. Filter the solution and wash with water to remove all impurities collect the precipitate and finally dried and weighed.

Recrystallisation:

It is recrystallised by using hot water.

Report:

The amount of salicylic acid is obtained =

Chemical reaction:

Ex.No:
Date:

PREPARATION OF UREA NITRATE

Aim:

To prepare and submit urea nitrate from urea

Principle:

The principle involved in the preparation of urea nitrate is nitration. Urea is nitrated with concentrated nitric acid to get urea nitrate

Chemical required:

Urea	-	2g
Conc. HNO_3	-	4ml
Distilled water	-	8ml

Procedure:

Take 2g of urea and dissolve in 8ml of distilled water and make into a saturated solution in a 100ml beaker and stirred well. To this add concentrated nitric acid drop by drop until the precipitate of urea nitrate is obtained. Wash the precipitate with concentrated nitric acid and finally dried and weighed.

Report:

The amount of urea nitrate is obtained =

Chemical reaction:

$H_2N\text{-}Co\text{-}NH_2 + HONO_2 \rightarrow H_2N\text{-}Co\text{-}NH_2 .HNO_3$

(Urea) (Conc. nitric acid) (Urea nitrate)

Ex.No:
Date:

PREPARATION OF BENZAMIDE

Aim:

To prepare and submit benzamide from benzoyl chloride

Principle:

Benzamide is an aromatic amide. It can be prepared by the action of benzoyl chloride with aqueous ammonia solution. It gives benzamide and hydrochloric acid. The formed hydrochloric acid is neutralized with excess of ammonia as ammonium chloride.

Chemical required:

Benzoyl chloride	-	5ml
Conc. ammonia solution	-	10ml

Procedure:

Take 10ml of Conc. ammonia solution in an iodine flask cool in a ice bath. To this add 5ml of benzoyl chloride gradually and shake the flask frequently until the precipitate of benzamide is obtained. Collect the precipitate and wash with water to remove all impurities and finally dried and weighed.

Recrystallisation:

It is recrystallised by using hot water.

Report:

The amount of benzamide is obtained =

Chemical reaction:

Ex.No:
Date:

PREPARATION OF ACETANILIDE

Aim:

To prepare and submit acetanilide from aniline

Principle:

The principle involved in the preparation of acetanilide is acetylation. Acetylation is done by using a mixture of acetic anhydride and sodium acetate or acetic anhydride and acetic acid. When aniline is treated with acetic anhydride and sodium acetate, it gets acetylated to give acetanilide.

Chemical required:

Aniline	-	3ml
Acetic anhydride	-	4ml
Sodium acetate	-	12.5ml
Conc.HCl	-	2.5ml

Procedure:

Take 100ml of water in a 250ml clean conical flask and add 2.5ml of Conc.HCl and shake well. Then add 3ml of aniline and stirred well until aniline completely passes into solution as anilinium ion. Add 4ml of acetic anhydride to the resulting solution until it dissolves completely. Add 12.5ml of 33% sodium acetate and stirred well vigorously cool the solution. It is then filtered and wash with water to remove impurities and finally dried and weighed.

Recrystallisation:

It is recrystallised by using hot water

Report:

The amount of acetanilide is obtained =

Chemical reaction:

Ex.No:
Date: PREPARATION OF BENZANILIDE

Aim:

To prepare and submit benzanilide from aniline

Principle:

Benzanilide is an aromatic anilide. The principle involved in the preparation of benzanilide is benzoylation. When treated with benzoyl chloride in the presence of aqueous sodium hydroxide to give benzanilide. The aqueous sodium hydroxide is used to remove benzoyl chloride which gives sodium benzoate and hydrochloric acid. This reaction is called Schotten Boumann method of benzoylation.

Chemical required:

Aniline	-	3ml
Benzoyl chloride	-	5ml
10% NaOH	-	4ml
Alcohol	-	5ml

Procedure:

Take 3ml of aniline and 40ml of 10% of sodium hydroxide in a 250ml of conical flask and mix well. To this add 5ml of benzoyl chloride and shake the flask continuously for 15-20 minutes,until the smell of benzoyl chloride ceases completely. Collect the precipitate and wash with water and finally dried and weighed.

Recrystallisation: It is re-crystallised by using alcohol.

Report:

The amount of benzanilide is obtained =

Chemical reaction:

Ex.No:
Date:

PREPARATION OF ACETYL SALICYLIC ACID

Aim:

To prepare and submit acetyl salicylic acid from salicylic acid

Principle:

Acetyl salicylic acid is an aromatic acid. It can be prepared when salicylic acid is treated with acetic anhydride or acetyl chloride in the presence of concentrated sulphuric acid. It gets acetylated to give acetyl salicylic acid

Chemical required:

Salicylic acid - 2g
Acetic anhydride - 4ml
Conc.H_2SO_4 - 3-4 drops

Procedure:

Take 2g of salicylic acid and 4ml of acetic anhydride in a 100ml clean dry conical flask. To this add 3-4 drops of Conc.H_2SO_4 and shake well for 15 minutes until the precipitate of acetyl salicylic acid is obtained. To this add 50ml of distilled water and filter the solution. Collect the precipitate and finally dried and weighed.

Recrystallisation:

It is recrystallised by using hot ethanol or boiling water.

Report:

The amount of acetyl salicylic acid is obtained =

Chemical reaction:

STEREO CHEMISTRY MODEL

Ex.No:
Date:

METHANE

Aim:

To make a stereo model of methane by using ball and stick molecules and explain the formation of bonds, bond angles and bond length.

Description:

Shape of the molecule	-	Tetrahedral
Bond angle	-	109^0 28’
Bond length	-	$1.09A^0$
Bond energy	-	102K Cal/ mole

(To break any one of the bonds of methane)

Hybridization:

Four Sp^3 orbital are found by the interaction of its one S orbital and three p orbital to give a four new equivalent orbital of identical (some energy and shape)

The four Sp^3 orbital are arranged in such a way that their axis are directed towards the corners of the regular tetrahedral with carbon located at the center.

Each carbon- hydrogen covalent bond in methane is formed by overlap of one Sp^3 orbital from carbon and one s from hydrogen.

Ex.No:
Date:

ETHYLENE

Aim:

To make a stereo model of ethylene by using ball and stick models and explain the formation of bonds, bond angles and bond length.

Description:

Bond angle	-	120^0
Bond length(C=C)	-	$1.34A^0$
Bond Length(C-H)	-	$1.09A^0$

Hybridization:

The ethylene, carbon atoms are Sp^2. The two Sp^2 hybridized carbons are attached to each other by σ bond and π bond. The σ bond results from the overlap of unhybridized p orbital. The remaining Sp^2 orbital form σ bond with either carbon (or) hydrogen atom. Ethylene is a planar molecule and is trigonal shape. The carbon-carbon double bond in alkenes is made of one σ bond and π bond.

Ex.No:
Date:

ACETYLENE

Aim:

To make a stereo model of acetylene by using ball and stick models and explain the formation of bonds, bond angles and bond length.

Description:

Bond angle	-	180^0
Bond length (C≡C)	-	$1.20A^0$
Bond length (C-H)	-	$1.09\ A^0$
Bond energy (C-C)	-	123-199 K Cal/ mole

Hybridization:

In this molecule the hybridization of one s- orbital and one p- orbital to form two equivalent collinear orbital which are directed in straight line with an angle of 180^0. The other two p- orbital remained pure.

In acetylene one of the Sp orbital of each carbon atom overlap with one another along the axis to form C-C σ bond. The remaining one Sp orbital of each carbon atom overlap with two s-orbital of hydrogen atom to form two C-H σ bond. The left pure p-orbital (py,pz) of each carbon atom containing electrons overlap to form two π molecule orbital. This π molecule orbital at right angle to each other and the result is a molecule in which π electron density cylindrically symmetrical about linear Sp bond.

Ex.No:
Date:

DETERMINATION OF MELTING POINT

Aim:

To determine the melting point of the given sample.

Principle:

The melting point is defined as the temperature of which the liquid and solid forms of a compound exist in its equilibrium with each other. The pure compounds have very short melting range where as impure compounds have wide melting point hence if a substance is not pure it will not melt over 0-5^{0}C range or less while the other compounds of ordinary purity will melt over at a range of 1-2^{0}C.

Procedure:

A small amount of the substance is powdered well in a clean porous plate with the help of spatula. A fine capillary tube of 8cm length is taken and sealed at one end by insulating in a Bunsen blue flame. The material is filled in the tube by pressing through the open end with sample kept in a watch glass and then tapping the closed end gently on a desk in order to force the sample down to the bottom. The process of tapping is repeated till the packed substance in the capillary tube stands 3-4mm from the sealed end. A 50ml glass beaker is half filled with liquid paraffin and then it is placed on wire gauze supported by tripod stand. A thermometer is kept front in a iron stand with the help of a cork it is covered into beaker, So that the bulb is well immersed in liquid paraffin the capillary tube which is with the substance is tied with thermometer. The tip of thermometer and capillary tube are in same level.

Now the beaker is slowly heated by means of Bunsen burner and the liquid paraffin is stirred gently at the same time watch the substance in the capillary tube and mercury level in the thermometer. As the substance shows the melting at once the burns is removed and stirring of both is continued. The temperature will rise very slowly when some of the substance in the capillary tube rises the temperature on the thermometer is noted.

The above determinations are repeated twice after cooling the bath and take fresh capillary tube each time. The mean of the three reading gives exact melting point of substance.

Note:

Don't take a large amount of liquid in a heating bath it will take a long time to heat and thus causes unnecessary delay.

At high temperature the stirring of bath becomes unnecessary because the conventional current that are set up heat the bath uniformly.

Sometimes sulphuric acid in the beaker become blackish, to decolourise the bath add potassium nitrate will oxidize the crystals before warming. The nitric acid produced will oxidize the colouring matter and the bath becomes colourless.

For cooling the bath liquid paraffin or sulphuric acid stir it in or mixed it with same fresh liquid of substance. Keep melting over a wide range of temperature it is impure. A pure substance must have sharp melting point.

The liquid paraffin bath should be free from water molecule otherwise the liquid may explode.

Report:

The melting point of given sample is =

Melting Point of some pure compounds

S.No	Compound name	Melting Point of pure compound in 0c
1	Benzoic acid	121
2	Salicylic acid	158
3	Acetanilide	114
4	Aspirin	135
5	Hydroquinone Diacetate	122
6	Iodoform	119
7	2,4,6- Tribromo Anilide	119
8	Para Bromo Anilide	167
9	Urea Nitrate	163
10	Picric Acid	162
11	5-NitroSalicylic acid	236
12	Benzanilide	162
13	2-Naphthyl Benzoate	115
14	Benzamide	129
15	Formic Acid	200

Ex.No:
Date:

DETERMINATION OF BOILING POINT

Aim:

To determine the boiling point of the given sample.

Principle:

A pure organic compound liquid boils at a fixed temperature which is characteristic of that substance.

Procedure:

When only a small quantities of liquid its boiling point is determined by the capillary tube method. A few drops of liquid are placed in a thin walled small test tube. A capillary tube sealed at about 1cm from one end is dropped on to it. The glass tube containing the liquid and capillary is then tied along the thermometer is then so that liquid stands near tube. The thermometer is then dipped in a beaker containing paraffin oil or sulphuric acid.

The beaker is heated and the bath liquid stirred continuously with using a stirrer when the boiling point is reached bubble starts in a rapid stream from the lower end of capillary tube .The thermometer is read .The evolution of bubbles is just stopped and the experiment is repeated .The mean of the two reading is taken to be correct boiling point.

Report:

The boiling point of given sample is =

S.No	Compound Name	BP in corrected	Degree in Celsius observation
1	Acetone	56	54
2	Aniline	183	180
3	Phenol	180	178
4	Benzene	80	82
5	Acetophenone	202	200
6	Phenyl Salicylate	224	222
7	Chloroform	51	48
8	Ethyl alcohol	78	76
9	Ethyl acetate	77	73

GENERAL SCHEME FOR IDENTIFICATION OF ORGANIC COMPOUNDS

Experiment	Observation	Inference
Preliminary test Physical State	Solid	May be acids ,amides
	Liquid	Phenols,alcohols,esters,aldehydes, ketones,nitro compounds
2.Colour	Colourless	Acids, aldehydes, Ketones,Esters,Hydrocarbons
	Yellow	Aromatic Nitro compounds
	Brown or black	Phenols ,amines
3.Smell(odour)	Pleasant Fruity	alcohols,esters
	Fishy or ammoniacal	Amines ,hydrocarbons
	Kerosene like	Hydrocarbons
	Pungent and irritating	Acids ,acid halides
	Bitter Almonds	May be benzaldehyde ,nitrobenzene
	Phenolic	Phenols
Solubility		
Cold water	Soluble	Sugars,acids,urea,thiourea Aromatic acids
Hot water	Soluble	Carboxylic acid
5%Sodium bicarbonate soln	Soluble with effervesence	Phenols , acids
5%sodium hydroxide	*Soluble and reappears on adding Hcl	Sugars ,Aliphatic aldehyde
	*Soluble and turns yellow or brown when boiled	
	*Soluble or oily layer disappears on boiling and forms white ppt on acidification with Hcl	Esters or amides of aromatic acid
Dil Hcl soln	Soluble	Amines

Con H_2So_4	Soluble	Esters
Litmus paper test		
Dissolve the substance and dip the litmus paper	Red to Blue	Amines
	Blue to red	Phenols or acids
Neutral Ferric chloride soln test		
Add one or two drops of Neutral Ferric chloride soln	Violet,blue ,green	Phenols
	White ppt	α Naphthols
	Green colour	β Naphthols
	Brown ppt or buff colour	Carboxylic acids
	Yellow	Hydroxy acids
Aromatic /Aliphatic		
1.Ignition test		
A small quantity of substance is ignited on a nickel spatula	Burns with Nonsmoky flame	Aliphatic compound
	Burns with smoky flame	Aromatic compound
2.Nitration test(Electrophilic substitution) To a little of substance add a mixture of 1ml of conc.HNO3 and 1ml of Con H2So4.It is then heated on a water bath for half an hour and poured into a beaker containing 25 ml of water	Colourless soln	Aliphatic compound
	Yellow colour soln	Aromatic compound
Saturated /Unsaturated		
1.Little of substance is shaken with water and add Bromine water	Yellow colour decolourised	Unsaturated
2.Little of substance is dissolved in Carbon tetra chloride and Bromine water is added in drops	Yellow colour persists	Saturated
	Yellow colour decolourised	Unsaturated

Baeyers test Little of substance is shaken with water and add one drop of Sodium carbonate solution and one or two drops of Dil Kmno4 solution	Yellow colour persists Pink colour decolourised Pink colour persists	Saturated Unsaturated Saturated
Test for Elements (Nitrogen,sulphur ,halogens) Sodium fusion test(Lassaigne's test) Add a small piece of freshly cut oil free sodium metal in a clean ignition tube . Heat it on the flame to melt it.Add little of the given sample ,heat it to red hot,and plunge immediately into about 10ml of distilled water in a china dish .Crush the fusion tube and boil the mixture for 5 minutes and filtered.This solution is called sodium fusion extract (SFE)		
Test for Nitrogen To 1ml of SFE in a test tube add few drops of freshly prepared ferrous sulphate powder.Heat it ,cool and acidify using Dil sulphuric acid Test for Sulphur 1.To 1 ml of SFE add one or two drops of freshly prepared	Prussian blue or green colour is formed Violet,red or purple colour	Presence of nitrogen Presence of sulphur

sodium Nitroprusside soln 2.To 1 ml of SFE add a drop of lead acetate solution	Black ppt	Presence of sulphur
Conformatory test for Carbohydrates Borsche's reagent A small quantity of 2,4 Dinitro phenyl hydrazine is moistened with con H2So4 and then add methyl alcohol and the given substance .Heat it in water bath	Yellowish orange colour	Presence of aldehyde or ketone
With Schiff's reagent	Violet colour	Presence of aldehyde
Sub+Tollens reagent(ammoniacal silver nitrate) and heat it	Silver mirror at the sides of testtube	Presence of carbohydrates
Sub+Fehlings reagent A&B,heat it in a water bath	Reddish brown ppt	Presence of Carbohydrates
Molisch test To the solution of the substance add alcoholic alpha naphthol and then add ConcH2SO4 along the sides of the test tube	Violet ring at the junction of two liquids	Presence of Carbohydrates
Osazone test Sub + water ,add phenyl hydrazine.Heat for 5 minutes in waterbath	Yellow ppt	Presence of Carbohydrates
Conformatory test for Carboxylic acid To the solution of the substance dip a glass rod and apply over Litmus paper	Blue to red	Presence of Carboxylic acid
Substance + sodium bicarbonate solution	Brisk effervescence	Presence of Carboxylic acid
Substance +little ethanol and a drop of Con.H2SO4.Heat it in	Fruity odour	Presence of Carboxylic acid

water bath for 5 miutes Fluorescein test Sub +equal quantity of resorcinol and moistened with a drop of Con H2SO4.Heat for 5 to 10 minutes and pour it into a beaker containing Sodium hydroxide soln	Intense greenish yellow fluorescence	Presence of Dicarboxylic acid
Conformatory test for Esters		
Hydrolysis Sub is refluxed with Concentrated solution of sodium hydroxide and then acidify with Con Hcl	White Ppt	Presence of esters
Hydroxamic acid test Sub+ 1 ml of Hydroxylamine hydrochloride in alcohol.To this add 1ml of sodium hydroxide and boil gently for 2 min.Acidify with dil hcl and add two drops of ferric chloride soln	Violet of deep reddish brown	Presence of esters
Conformatory test for Phenols		
Ferric chloride test Sub +Alcoholic or aqueous 5%ferric chloride soln	Blue or violet Pink or white Green to white Yellow	Phenol,resorcinol Alpha naphthol Beta naphthol Phenolic acid
Liebermann reaction Sub in a dry test tube add 1 ml of Conc H2SO4 ,few sodium Nitrite crystals heat it in waterbath ,pour into beaker having sodium hydroxide soln	Blue green or blue violet colour	Phenols
Phthalein test Heat the substance with phthalic anhydride and a drop		

of Con H2SO4 for 2 minutes .Cool and pour the contents to sodium hydroxide solution	Green Faint green	Phenols Alpha or beta naphthols
Bromine water is added to dil aqueous soln of phenols	Silky white ppt	Phenols
Sub +1ml of dil NaoH solution and one drop of CHCL3,warm the mixture	Blue colour	Alpha or beta naphthols
Differentiation for alpha and beta naphthols		
Warm with pinch of substance ,Carbon tetra chloride and copper powder	Blue colour Brownish colour	Alpha naphthols Beta naphthols
Sub + alcohol and add picric acid	Reddish pink Orange colour	Alpha naphthols Beta naphthols
Conformatory test for Amines		
Sub + dil Hcl	Soluble and gets precipitated on adding sodium hydroxide	Amines
Nitrous acid test Sub +Con Hcl cool it in ice bath.Add a cold solution of Sodium nitrite	Bubbles of nitrogen gas	Amines
Dye test Sub +dil Hcl,cool and add Sodium nitrite solution ,then add cold solution of Alkaline betanaphthol solution	Orange red dye	Amines
To the substance solution add bromine water	White ppt	Amines
Sub +glacial acetic acid and 0.5 ml of acetic anhydride heat it gently and pour into water in a beaker	White crystals	Amines

Conformatory test for Amides		
Heat the sub with sodium hydroxide solution	Ammonia gas is evolved	Amides
Heat the sub + Con. HNo3	White ppt	Amides
Sub + oxalic acid soln	White ppt	Amides
Biuret test A little of sub is gently heated in a dry testtube to melt it.add 1 ml of sodium hydroxide soln cool and add 2 drops of copper sulphate solution	Violet colour	Diamides
Sub + aniline heat it and cool	White ppt	Amides
Conformatory test for Thiourea		
Ferric chloride test Sub in a dry test tube is heated ,add dil sodium hydroxide soln ,acidify with dil Hcl,add drop of ferric chloride solution	Deep red	Presence of Thiourea
Warm the sub with alcoholic yellow mercuric oxide in a test tube	Black ppt	Presence of thio urea
Sub + 1 ml of dil acetic acid ,heat it,add 1 ml of potassium ferro cyanide solution	Green colour changing to blue	Presence of thio urea