

P.G. & Research Department of Chemistry

Islamiah College (Autonomous),

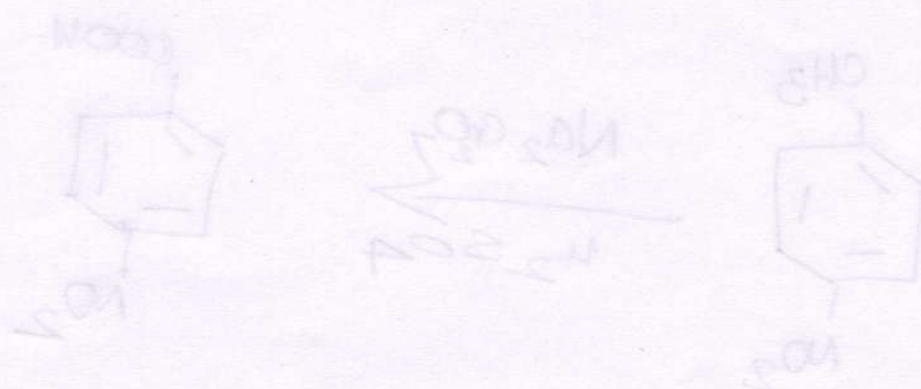
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Lab Manual for M.Sc. Chemistry

PRACTICAL ORGANIC CHEMISTRY

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PREPARATIONS

1. PREPARATION OF p-NITRO BENZOIC ACID FROM p-NITRO TOLUENE

Aim:

To prepare the p-Nitro benzoic acid from p-Nitro toluene.

Chemicals Required:

| | |
|------------------------|-------|
| ➤ P- Nitro toluene | 5 g |
| ➤ Sodium dichromate | 14 g |
| ➤ Conc. Sulphuric acid | 20 ml |

Procedure:

Place about 5g of p-Nitro toluene 14g of crystalline Sodium dichromate are fitted with a mechanical stirrer. By means of dropped funnel supported above the flask. Add 20ml of Conc. H_2SO_4 during about 30min to the will stirred mixture. The heat of dilution of acid causes the p-Nitro toluene to melt and oxidation takes place. If the reaction shows signs of becoming vigorous the rate of addition must be reduced. When all the H_2SO_4 has been introduced and temperature of the mixture commence to fall stirred the reflux condenser to the flask and heat to gentle boiling for half an hour, cool and pour the reaction mixture into 250ml water. Filter the crude p-Nitro benzoic acid at the pump and wash it about 200ml of H_2O . Transfer the solid to a 400ml beaker add about 20ml of 5% H_2SO_4 and digest in a water bath with agitation in order to remove the chromium salts as completely as possible allow it to cool and filter again to the beaker and treated with 5% NaOH solution until the liquid remains alkaline. P-Nitro benzoic acid pass into a solution any unchanged. P-

Nitrotoluene to remains undissolves and chromium salts are converted into Sodium dichromate.

Add about 1g of decolourising carbon warmed to about 50°C with stirring for 5 min and filter with suction pump, alkaline solution of p-Nitro benzoic acid into about 50ml of 15% H_2SO_4 do not add the acid into alkaline solution for the way. The acid is to liable to contaminated by the sodium salt. Filter the purified acid at the pump wash it in cold water and dry it in the steam oven, this is sufficiently pure for most purpose upon recrystallisation from benzene (or) glacial acetic acid.

Result:

The yield of p-Nitro benzoic acid is _____g

The Melting point of substance is 239°C

2. PREPARATION OF ANTHRO QUINONE FROM ANTHRACENE

Aim:

To prepare the anthro quinone from anthrecene

Chemicals Required:

| | |
|-----------------------|-------|
| ➤ Anthrecene | 5 g |
| ➤ Glacial acetic acid | 60 ml |
| ➤ Chromium trioxide | 10 g |
| ➤ Sodium hydroxide | |

Procedure:

About 5g of finely powdered pure Anthrecene in 60ml of glacial acetic acid contain in a 250ml of round bottom flask provided with a reflux condenser. Prepare a solution of 10g of Chromium trioxide in 15ml of water and add 30-40 ml of Glacial acetic acid and the Chromium trioxide solution slowly in 1hour to the boiling anthracene solution by means of a separating funnel. Fitted into the top of the condenser with a cork. Boil further 15 min allow the drop solution to cool and poured into 300ml of cold water.

Fitted the crude anthroquinone by suction pump a with a little hot water the by finally with cold water until the wash in are colourless and dried well. Recrystalised from boiling Glacial acetic acid. Little of the purified crystals with a little alcohol and dried at $100 - 200^{\circ}\text{C}$.

Result:

The yield of Anthroquinone is _____ g

The Melting point of Anthroquinone is 286°C

3. PREPARATION OF BENZYHYDROL FROM BENZOPHENONE

Aim:

To prepare the benzyhydrol from benzophenone.

Principle:

Benzhydrol is obtained by the reduction of benzophenone in the presence of base.

Chemicals Required:

| | |
|-------------------------|------|
| ➤ Benzophenone | 5 g |
| ➤ Sodium hydroxide | 5 g |
| ➤ Zinc powder (or) dust | 5 g |
| ➤ Rectified spirit | 50 g |

Procedure:

About 5g of benzophenone, 50ml of rectified spirit, 5g of zinc powder and 5g of Sodium hydroxide are taken in a 500ml Round bottom flask with reflux condenser and a mechanical stirrer, place 5ml of Benzophenone 50ml of rectified spirit and 50g NaOH and 5g of Zinc dust stir the mixture the temperature slowly raises to 70% , after 3 hrs the reaction mixture with suction and wash the residue twice with 25ml portion hot alcohol do not allow the residual Zinc dust to become dry as it is inflammable pour the filter onto 200ml

of ice water acidified with 10ml of Con HCL. The benzhydrol separate as white crystalline mass filter at a pump and dry in a air the yield of crude benzhydrol melting point 65°C is 5g recrystalline from 5ml of hot alcohol and cool in a freezing mixture of ice and salt collect the colourless crystals and dry in air. 3.5g of diphenyl carbinol melting point 68°C are obtained precipitate the mother liquor with water to recover the residual benzhydrol and recrystalline these from hot water.

Result:

The Yield of benzyhydrol is _____g

The Melting point of benzyhydrol is 65°C

4. PREPARATION OF METHYL ORANGE FROM SULPHONILIC ACID

Aim:

To prepare the Methyl orange from sulphonilic acid.

Chemicals Required:

| | |
|------------------------------|-------|
| ➤ Sulphonite acid dehydrate | 5g |
| ➤ Sodium nitrite | 1.8 g |
| ➤ Anhydrous Sodium carbonate | 1.3 g |
| ➤ Sodium hydroxide | 20 ml |
| ➤ Dimethyl aniline | 3 ml |
| ➤ Conc. Hydrochloric acid | 6 ml |

Procedure:

In a 250ml of conical flask place 5g of Sulphonite acid dihydride and 1.3g of anhydrous sodium carbonate and 100ml water and warmed until clear solution is obtained cool water under with 1.8g sodium nitrite in 10ml of water.

Pure resulting solution it is stirring into 50ml beaker containing 6ml of Conc. Hydrochloric acid and add ice. Test for free nitrous acid with potassium iodide starch paper. Fine crystals of Diazobenzene sulphonate will separate after 15 min.

Do not filter as this will be dissolved during next stage of preparation. Dissolved 3ml of N-N-Dimethyl aniline in 3ml glacial acetic acid and it with vigorous stirred to the suspension of diazotised sulphonilic acid.

Allow to stand to 10 min. The red orange from methyl orange will separate. Then add slowly with stirring 20 ml of 20% Sodium hydroxide solution. Then the separation of mixture assumes uniform orange colour due to the separation of fine crystals of methyl orange.

Direct filtration of later is shown hence this stirring the mixture with thermometer heat it almost to the boiling point most of the methyl orange will dissolve and add 5 g of Sodium chloride and warm 80-90⁰ C until the salt solution dissolves. Allow it to cool and undisturbed for 15 min and cool in ice bath. This gives easily filterable product. Filter of Methyl orange from pump rinse with beaker saturated solution and dry well. From hot water in crude sample is recrystallised. Filter it if necessary. Reddish orange crystal of Methyl orange will separate filter and dry the Methyl orange.

Result:

The yield of Methyl orange is _____ g

The Melting point of Methyl orange is 239⁰ C

5. PREPARATION OF O-BENZYL BENZOIC ACID

Aim:

To prepare the O-Benzyl benzoic acid.

Chemicals Required:

| | |
|---|------|
| ➤ Phthalic anhydride | 5g |
| ➤ Sodium- dried A.R (or thiophene-free) | 20ml |
| ➤ Anhydrous aluminium chloride | 2g |
| ➤ Anhydrous sodium carbonate | 2.6g |

Procedure:

Place 5g of pure phthalic anhydride and 20ml of sodium-dried A.R. (or thiophene-free) benzene in the flask, start the stirrer and add 10g. Of powdered anhydrous aluminium chloride from a stoppered test tube in a four portions. If the reaction does not commence after the addition of the first 2g. Of aluminium chloride, from the stoppered test tube. If the reaction does not commence after the addition of the first 2g. Of aluminium chloride, warm for a few seconds on a water bath. When all the aluminium chloride has been added and the evolution of gas practically ceases. Cool the flask add crushed ice slowly until the dark mass is completely decomposed, and then run in Con. Hydrochloric acid (7-8ml) until the solution clears. Steam distil to remove the excess benzene, the residue in the flask, when cooled in ice, largely solidifies and consists of crude of O-Benzyl benzoic acid., decant the aq. Solution through a Buchner funnel, wash the residue decantation with a little cold water, and return the solid, collected on the filter, to the flask. Add a warm solution of 2.6g of anhydrous sodium carbonate in 40ml of water and pass steam into the

mixture until all the solid material except particle of aluminium hydroxide and a little tarry material dissolve. Allow to cool slightly and add a few grams of decolourising carbon. Warm and filter the hot solution with suction, place the filtrate in a beaker, cool in ice and cautiously acidify with Con. HCl. While stirring and cooling, filter when ice cold and wash with a little water. Dry in the air upon filter paper the product which is somewhat efflorescent consists largely of the monohydrate m.p 94°C .

To prepare pure anhydrous O-benzyl benzoic acid dissolve the air-dried product in about 17ml of benzene contained in a 250ml R.B. Flask fitted with a reflux condenser and heat on a water bath. Transfer the benzene solution to a separatory funnel run off any water present and dry with anhydrous magnesium sulphate. Concentrate benzene solution to about 7.5ml and light petroleum ($60-80^{\circ}$) to the hot solution until the slight turbidity is produced. Allow to cool spontaneously to room temperature, then cool in ice to about 5°C , collect the crystals and dry. The yield of pure, anhydrous O-benzyl benzoic acid.

Result:

The yield of O-Benzyl benzoic acid is _____ g

The Melting point of O-Benzyl benzoic acid is 128°C

6. PREPARATION OF m- NITRO ANILINE FROM m-DINITRO BENZENE

Aim:

To prepare the m- Nitro aniline from m-dinitro benzene.

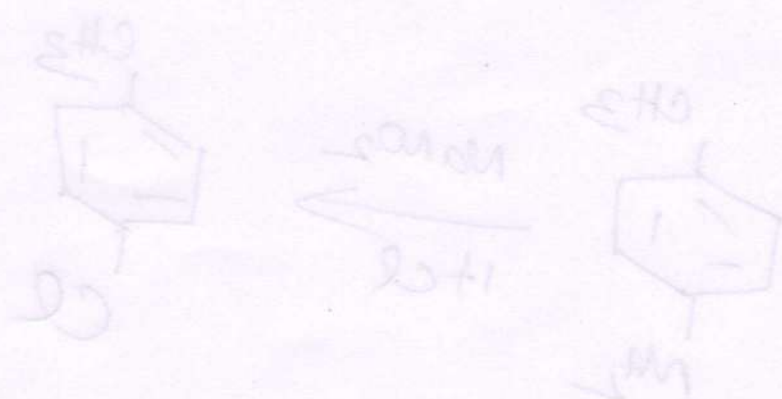
Chemicals Required:

| | |
|--------------------------------|------|
| ➤ Sodium poly sulphide | 10g |
| ➤ Crystallised sodium sulphate | 35ml |
| ➤ Sulphur | 2.5g |
| ➤ m-Dinitro benzene | 6.2g |
| ➤ Conc. Hydrochloric acid | 8ml |

Procedure:

Prepare a solution of Sodium polysulphide by dissolving 10g. Of crystallised sodium sulphate in 35ml, of water adding 2.5g of finely powdered sulphur, and warming until a clear solution is produced. Heat a mixture of 6.2g, of m-dinitro benzoic acid and 40ml of water contained in a 1 litter beaker until the water boils gently, stir the solution mechanically. Place the sodium polysulphide solution in a dropping funnel and clamp the funnel so the end of the steam is immediately above the beaker. Add the polysulphide solution during 30-45min to the vigorous stirred, boiling mixture and boil gently for the 20min, allow to cool. This can be accomplished more rapidly by adding ice, filter at the pump and wash with cold water. Transfer to a 400ml beaker containing 40ml of water and 8ml of Con.HCl, and boll for 15min, the m-nitro aniline dissolves leaving the sulphur and any unchanged m-dinitro benzene.

Filter and precipitate the m-nitro aniline from the filtrate by addition of excess of concentrated aq. Ammonia solution. Filter off the product and recrystallised it from boiling water.



Result:

The yield of m-Nitro aniline is _____ g.

The Melting point of m-Nitro aniline is 114°C

7. PREPARATION OF P-CHOLORO TOLUENE FROM P-TOLUIDENE

Aim:

To prepare the p- Chloro toluene from p- toludene.

Chemicals Required:

| | |
|--------------------------|-------|
| ➤ p- Toludene | 7 g |
| ➤ sodium nitrite | 5 g |
| ➤ Con. Hydrochloric acid | 17 ml |
| ➤ Sodium hydroxide | 6 ml |
| ➤ Diazonium chloride | |

Procedure:

Dissolve 7g of p-Toludene in 17ml of Con. HCl and 17ml of water contained in a 500ml conical flask. Cool the mixture to 0°C in an ice salt bath with vigorous stir or shaking and addition of a little crushed ice. The salt, p-toludene hydrochloride will separate as a finally divided crystalline precipitate add during 10-15min of solution 5g of sodium nitrite in 10ml of water shake the solution well during the diazotisation and keep the mixture at a temp, $0-5^{\circ}\text{C}$, by the addition of a little crushed ice from time to time. The hydrochloride will dissolve as the very soluble diazoinun salt is formed. When all the nitrite solution has been introduced the solution should contain the trace of solution has been introduced, the solution should contain a trace of free nitrous acid.

Pour the cold diazonium chloride solution slowly and with shaking into the cuprous chloride solution. The mixture becomes very thick, owing to the separation of an addition product between the diazonium salt and cuprous

chloride. Allow the mixture to warm up to room temperature without external heating and shake occasionally. When the temp reaches about 15° the solid addition complex commences to break down with the liberation of nitrogen and the formation of an oily layer of p-chloro toluene. Warm the mixture on water bath to about 60° to complete the decomposition of the double salt, shake occasionally. When the evolution of nitrogen ceases, steam distil the mixture until the no more oily drops are present in the distillate. Transfer the distillate to the separate funnel, and remove the layer of p-chloro toluene. Wash with 30ml of 10% NaOH solution (to remove any p-creosol which may be present) water, an equal volume of Con. H_2SO_4 (to remove trace of azo compound that usually colours the crude product and cannot be removed distillation) and water (to remove the acid. Dry with 3-4g of anhydrous calcium chloride or any anhydrous magnesium sulphate, decant or filter through a small fluted filter paper into a small distilling flask and distil on asbestos centred gauze or from an air bath. Collect the p-chloro toluene.

Result:

The yield of p-Chloro toluene is _____ g.

The Melting point of p-Chloro toluene is $158-162^{\circ}\text{C}$

8. PREPARATION OF DIPHENYL METHANE FROM BENZYL CHLORIDE

Aim:

To prepare the Diphenyl methane from benzyl chloride.

Chemicals Required:

| | |
|--------------------------------|-------|
| ➤ Benzyl chloride | 7.7 g |
| ➤ Dry benzene | 30 ml |
| ➤ Anhydrous aluminium chloride | 2.4 g |

Procedure:

Place 7.7g (7ml) of redistilled Benzyl chloride and 30ml of dry benzene in the 500ml R.B.Flask. Weigh out 2.4g of anhydrous aluminium chloride into a dry-stoppered test tube with minimum exposure to the atmosphere. Cool the flask in a bath of crushed ice and add about one fifth of aluminium chloride. Shake the mixture. A vigorous reaction will set in with few minutes and hydrogen chloride will be evolved. When the reaction has subsided, add further portion of aluminium chloride and repeat the process until all has been introduced. The mixture should be kept well shaken and immersed in a freezing mixture during the addition. Finally reflux the mixture on a water bath for 30min. Allow to cool, cautiously add 20g of crushed ice, followed by 20ml, of water in order to decompose the aluminium complex. Shake the mixture well, transfer to a separatory funnel, and run off the lower aq. Layer. Wash the upper layer (benzene solution of diphenyl methane etc,) successively with Dil. Hydrochloric acid and water. Dry the benzene solution with anhydrous calcium chloride (warming on a water bath is disadvantageous). Remove the with aid of the apparatus. Distil the remaining liquid through an air condenser

either with free flame or from an air bath. Collect the Diphenyl methane at 250-275⁰ (the pure substance boils at 262⁰). The distillate should solidify on cooling in ice and scratching with glass rod, or by seeding with a crystal of pure material. If it does not crystallise, redistill from a small flask and collect the fraction of boiling point 255-267⁰ C. This generally crystallises on cooling and melts at 24-25⁰ C.

Result:

The yield of Diphenyl methane is _____ g.

The Melting point of Diphenyl methane is 24-25⁰ C

SEMI MICRO
QUALITATIVE
ANALYSIS

SEMIMICRO QUALITATIVE ANALYSIS

A. PRELIMINARY REACTION

| No: | Experiment | Observation | Inference |
|-----|---|--|---|
| 1. | Colour: Observe the colour of substance | i) Yellow ii) Dark brown iii) Colour less | Aromatic nitro compound, quinone, diketone. Aromatic amine, phenol Acid, aldehyde, ketone, ester, carbohydrate. |
| 2. | Smell: Note the smell of sub. | i) Pleasant or fruity ii) phenolic iii) aniline like iv) kerosene like v) pungent irritating | Ester Phenol Aromatic amine Hydrocarbon Aromatic comp. With halogen in side chain. |
| 3. | Litmus test: 0.1g (2dp.) Sub. +2ml. Water. Shake well and test Soln. With litmus paper. | Blue turns to red. Red turns to Blue | Acid or phenol Amine. |
| 4. | Solubility tests: 0.1g sub. +2ml. Satd. NaHCO_3 Soln. | Dissolves with brisk effervescence of CO_2 . | Acid and Acid chloride |
| 5. | 0.5g Sub. + 2ml dil.HCL. Shake well. | Dissolves in HCL, reappears on adding 10%NaOH drop wise. | Basic substance. (Amine) |
| 6. | 0.5g sub. + 2ml 10%NaOH. Shake well | Dissolves in NaOH, reprecipitations on adding Con.HCL. | Aromatic acid, Phenol |

| | | | |
|----|---|--|--|
| | | <p>Dissolves producing a strong yellow colour, which is removed by Conc. HCL.</p> <p>Ammonia gas evolved.</p> <p>Solution turns yellow to brown when boiled.</p> <p>Oily globules obtained having aniline like smell, when boiled.</p> <p>Oily layer disappears on boiling; white ppt. Obtained on acidification with Conc.HCL. and cooling.</p> <p>The sub. Unaffected in cold and on boiling</p> | <p>Nitro phenol, salicylaldehyde.</p> <p>Ammonium salts, amide</p> <p>Sugar, aliphatic aldehyde.</p> <p>Aniline.</p> <p>Ester and amide of aromatic acid.</p> <p>Nitro comp. Ketone, amine, hydrocarbon, aromatic halogen compd.</p> |
| 7. | <p>Action on Con. H_2SO_4: 0.1g sub. + 1ml Con.H_2SO_4. warm gently.</p> | <p>Sub. Becomes black with burnt sugar smell.</p> <p>Violent reaction.</p> | <p>Carbohydrate, polyhydric alcohol.</p> <p>Aliphatic or aromatic alcohol.</p> |
| 8. | <p>Soda lime test: 0.1g sub. + 1g soda lime. Heat mixture strongly.</p> | <p>Ammonia gas liberated.</p> <p>Kerosene smell</p> <p>Phenolic smell.</p> | <p>Ammonium salts or, amide</p> <p>Acid</p> <p>Phenolic acid, ester.</p> |
| 9. | <p>Neutral $FeCl_3$ test: 0.1g sub + 1ml. Water (or alcohol if insoluble in water) + 5dp.* neutral $FeCl_3$.</p> | <p>Violet, blue or green colour.</p> <p>Blue ppt.</p> | <p>Phenol.</p> <p>Naphthyl amine, Naphthol.</p> |

| | | | |
|-----|---|------------------------------------|--|
| 10. | Tollens reagent: 0.1g sub. +2ml. Tollens reagent. Heat in water bath. | Brown ppt. | Carboxylic acid. |
| | | Strong yellow colour. | Hydroxyl acid. |
| | | Black ppt. Or bright silver mirror | Aldehyde, reducing sugar, polyhydric alcohol, or other reducing agent. |

B. TESTS FOR SATURATION / UNSATURATION

| | | | |
|-----|---|---|--|
| 11 | 0.1g sub. + 1ml water +3-4dp. Bromine water. Shake well. | Yellow colour decolourised. | Unsatd. Compd. |
| | | Yellow colour persists. | Satd. Compd. |
| | | Yellow colour decolourised, ppt formed. | Satd. Compd. Like amine, phenol. |
| 12. | 0.1g sub + 3-4dp. Bromine in CCL ₄ (instead of Br ₂ /H ₂ O, if sub. Does not dissolve in water). Shake well. | Yellow colour decolourised. | Unsatd. Compd. |
| | | Yellow colour persists. | Satd. Compd. |
| 13. | 0.1g sub. + 1ml. Na ₂ CO ₃ soln. + 1% KMnO ₄ drop wise. | Pink colour decolourised. | Unsatd. Compd. Easily oxidisable compd. (aldehyde) |
| | | Pink colour remains. | Satd. Compd. |

C. TESTS FOR ALIPHATIC / AROMATIC NATURE

| | | | |
|-----|--|----------------------------|------------------|
| 14. | Ignite 0.1g.(2dp) sub. In Ni spatula. | Burns, sooty flame. | Aromatic compd. |
| | | Burns, non-luminous flame. | Aliphatic compd. |
| | | Yellow ppt or yellow soln. | Aromatic compd. |
| 15. | 0.2g sub. + a mix. Of 1ml Con.HNO ₃ and 1ml con. H ₂ SO ₄ . Shake well. Heat mixture for 10-15 min on a boiling water bath. Pour it into 20 ml of cold water. | No yellow colour. | Aliphatic compd. |

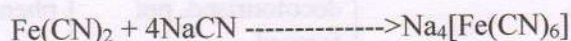
D. TESTS FOR ELEMENTS

16. Sodium fusion tests:

Melt a small piece of dry Na in a small fusion tube. Add 0.1g. Solid (or 3dp.liquid) to fusion tube. Heat gently at first, then to red-hotness. Quickly and carefully plunge red-hot end of tube into 10ml. Distilled water in china dish. Well with broken end of tube, boil and filtrate into 4 parts and test as follows:

TESTS FOR NITROGEN:

1ml. Fusion extract + few crystals of ferrous sulphate. Boil and cool. Add 2ml. Dil. H_2SO_4 . Blue/ green ppt. or soln. Indicates **Nitrogen**.

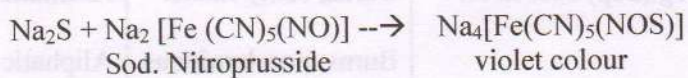
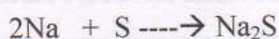


Fe^{3+} in the last step originates from aerial oxidation of Fe^{2+} of FeSO_4 soln. (on standing).

TESTS FOR SULPHUR:

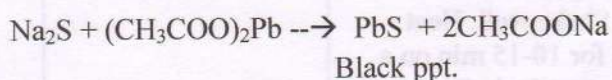
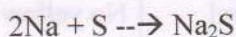
Sodium nitropruside test:

To 1ml. Fusion ext. Add 1ml. Of sod. Nitropruside soln. Violet colour shows **Sulphur**.



Lead acetate test:

To 1ml. Fusion ext. Add 1ml, of lead acetate soln. Black ppt. Shows **Sulphur**.

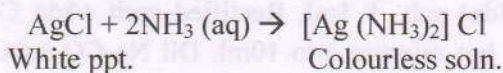
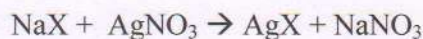
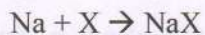


Test for Halogens:

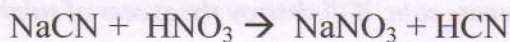
1ml. Fusion ext. + 1ml.dil.HNO₃. Boil, and then cool. Add ml. AgNO₃. White ppt. (easily soluble in aq.NH₃) shows **Chlorine**,

Pale yellow ppt. (partially soluble in aq.NH₃) shows **Bromine**.

Deep yellow ppt. (insoluble in aq.NH₃) shows **Iodine**.

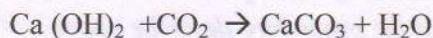
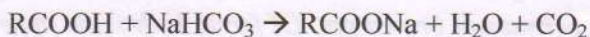


Note: -- the ext. should be boiled well with dil.HNO₃ (especially when N is present) to destroy NaCN, which would interfere by forming insoluble white ppt, of AgCN with AgNO₃.

**E. TESTS FOR FUNCTIONAL GROUPS****1. TESTS FOR ACIDS:**

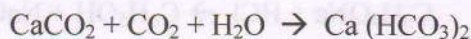
Litmus test: 0.1g sub (3dp). +1ml water, shake well, add well dp of this soln. To blue litmus paper if it turns Red, an acid is present.

NaHCO₃ test: To 0.1g sub. (3dp). Add 1ml of satd. NaHCO₃ soln. A brisk effervescence with the evolution of CO₂. (This turns limes water milky shows as acid).



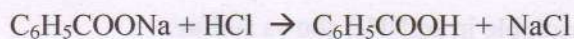
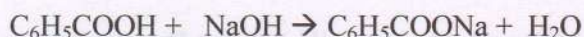
White ppt

Note: -- on passing carbon dioxide into lime water for a long time, the white ppt., formed initially, dissolved (due to formation of calcium bicarbonate).



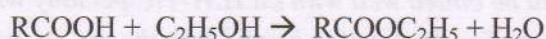
Colour less soln.

NaOH test: 0.1 g sub (3dp). +1ml of 10% NaOH soln. Shake well. All organic acids dissolve when NaOH. On acidifying acids with conc. HCl, the acid is ppt. If it is insoluble in water (aromatic acid),



Note: -- if the given acid is soluble in water (e.g., lower aliphatic acid), precipitation will Not takes place.

Etherification: 0.1g (3dp) sub. + 1ml. Rectified spirit +5dp Con. H_2SO_4 . Shake well and warm gently. Pour the hot mixture into 10ml. Dil Na_2CO_3 soln. Pleasant odour (due to the formation of an ester) indicates an organic acid.



(Note: -- Sodium carbonate soln. Will destroy the excess of sulphuric acid)

Phenolphthalein (Hph) test: Dissolve 0.1g (3dp) of sub. In 1ml water. Add a dp. Of Hph then add dil. NaOH, drop by drop, pink colour after 2 or 3 dp shows acid.

NaOH first neutralises the organic acid, then its excess gives pink colour with Hph.

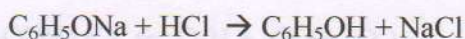
Fluorescence test: Mix 0.1 g.sub. With 0.5g resorcinol and 4dp. Con. H_2SO_4 in a heard for 3-5mts. Pour into dil.NaOH. An instance greenish yellow fluorescence indicates a dicarboxylic acid which forms an inner anhydride (e.g. Succinic acid, phthalic acid.)

2. TESTS FOR PHENOL:

Neutral Ferric Chloride test: 0.1g (3dp) sub. + 5dp neutral ferric chloride soln. Appearance of blue violet or green colour indicates a phenol. The colour is due to complex formation between ferric ion and phenol (or substituted phenol).

Note:- Prepare neutral ferric chloride soln. As follows. Too 1ml ferric chloride soln. Add aq. Ammonia, till a permanent brown ppt. Of ferric hydroxide appears. Add dil. HOAc drop wise, till the ppt. Just dissolve. If solid ferric chloride is available, dissolve 0.5g in 50ml. Water + 2dp.dil.HCl repeat the procedure describe above

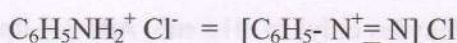
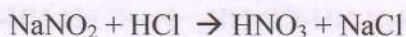
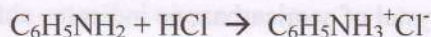
NaOH test: shake 0.1g (3dp) sub. With 1 ml 10% NaOH soln. The sub. Dissolve and reappears on adding Con. HCl, showing the presence of phenol, (Test is common for aromatic acid also.)



Note: - Phenol do not give CO_2 with NaHCO_3 soln. This test could be used to differentiate phenols from acids

Phthalic anhydride test: 0.1g. (3dp) sub. + 0.1g phthalic anhydride + 3dp. Con. H_2SO_4 . Heat gently for 3-5 min and pour into 20ml .dil. NaOH soln. Appearance of pink colour (due to phenolphthalein) indicates monohydric phenol; greenish yellow colour (due to fluorescence) indicates dihydric phenol (e.g., resorcinol).

Dye test: dissolve 1ml. aniline in 4ml. Dil. HCl . Cool the soln. To this soln. (which contains diazonium salt) add slowly 0.5g sub. Dissolved in 5ml. Dil. NaOH soln. Orange dye shows phenol, scarlet red dye shows naphthol, resorcinol, etc.



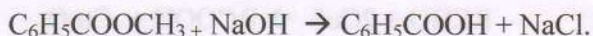
Liebermann's test: mix 0.1 g. Sub. With 0.5g NaNO_2 in a dry test tube, heat for 2mts. Cool. Add 5dp. Con.sulphuric acid. A blue green soln. Is got. Pour this into 30ml. Water in a beaker, and add NaOH in excess. A red soln. obtains first, with excess NaOH , the colour changes to deep blue.

3. TESTS FOR ESTER:

Hydrolysis: 0.2g sub. + 2ml.10% NaOH . Boil for 3 mts, the disappearance of pleasant odour indicate ester.



Precipitation of acid: cool the reaction mixtures from above test add 1ml. Con. HCl , a white ppt. Indicates aromatic ester.



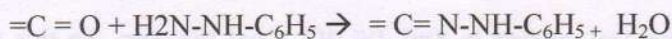
Tests with phenolphthalein: 0.2g. Sub. + 2dp. Phenolphthalein + 3dp. NaOH soln. Heat pink colour disappearance on heating indicates an ester.

Initially the free NaOH gives pink with indicator. On heating, NaOH is used for

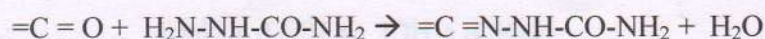
4. TESTS FOR ALDEHYDES AND KETONES:

Borsches test: To 0.1g sub. Add 1ml. Borsches reagent. Boil and add con.HCl. cool, add 1ml water, yellow or orange ppt. Shows aldehyde or ketone.

Phenyl hydrazine test: 0.1g sub. +1ml. Ethanol + 1ml phenyl hydrazine. Heat in a boiling water bath for 10 mts. Yellow ppt. Indicates aldehyde or ketone. Deep yellow osazone indicates aldoses (like glucose)



Semicarbazide test: 0.2g sub. + a mixture of 0.2 g semicarbazide hydrochloride, 0.5g sod. Acetate and 2ml water. Add about 1ml. Of alcohol, drop by drop to get clear soln. Heat for 2mts. Cool, a colourless crystal of semicarbazone indicates aldehyde or ketone.



Schiff's reagent test: (aldehydes only) to .01g sub. Add 3-4 dp. Schiff's reagent. Shake well. Immediate pink or red colour shows aldehyde (some aliphatic ketenes, like acetone also give a pink colour, but very slowly).

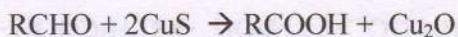
Schiff's reagent is produced from p-rosaniline hydrochloride.

The pink colour of this soln. In water is decolourised by passing sulphur dioxide. This decolourisation results from destruction of quinonoid structure of the ye by H_2SO_3 . Aldehyde structure, hence pink colour.

Fehling's soln. Test: For aliphatic aldehyde only). To 0.1g sub. Ad 1ml. Each of Fehling's soln. "A" and "B". Heat to boil. A red ppt. shows aliphatic aldehyde or aloes (like glucose).

Fehling's soln. "A": Copper sulphate soln.

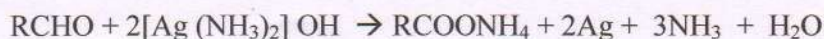
Fehling's soln. "B": Alkaline sod. Pot. Tartarate.



Cuprous oxide (red ppt.)

Tollens reagent test: (aldehydes only). To 0.1g.sub. add 1ml. Tollens reagent. Heat the test tube in a water bath for 5-10 mts. Black ppt. (or bright silver mirror) indicates aldehyde or aldose (like glucose).

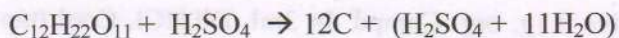
Tollens reagent: Ammonical $AgNO_3$ Soln.



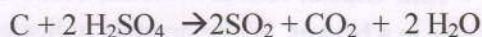
Legal test: (for ketones only) 0.1g.sub. + 2ml. Water +5p. Sod. Nitroprusside soln. + 5dp NaOH + 5dp. Glacial acetic acid. Oran colour to purple, indicates ketone,

5. TESTS FOR CARBOHYDRATES:

Charring test: To 0.1g. Sub. In dry test tube, add 5dp. Con.Sulphuric acid. Heat gently. Charring (with burnt sugar smell) indicates carbohydrates.



Carbohydrates lose water when heated with Con.Sulphuric acid. Sulphur dioxide is evolved.



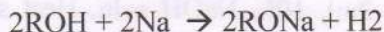
Molish test: 0.1g. Sub. In 1ml water + 1ml. Alcoholic α -naphthol soln. Shake well. Add Con. H_2SO_4 , drop by drop, through side of the test tube. The formation of violet ring at the junction of the two liquids, and the separating of colour on standing, shows carbohydrate.

The violet ring may be due to an unstable condensation product of α -naphthol with furfural. (Formed by the cyclodehydration of a carbohydrate with acid.

Osazone test: 0.1g sub. In a 3ml.water + 1mlphenylhydrazene. Heat for 5-10mts. In a boiling water bath. Yellow crystals of osazone shows monosaccharide (like glucose).

6.TESTS FOR ALCOHOL:

Na test: 0.5ml. org. Liquid + 1ml benzene + small piece of sodium. Vigorous reaction with evolution of Hydrogen. (pop sound with burning splinter) indicates alcohol.



Esterfication: 0.1g sub. + 1ml acetic anhydride + 3dp.Con. H_2SO_4 . Heat for 2-3mts. Cool pour into 20 ml. Dil. Na_2CO_3 soln.



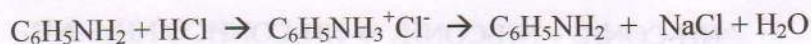
HOAc is neutralised by Na_2CO_3 soln.

TEST FOR N COMPOUNDS

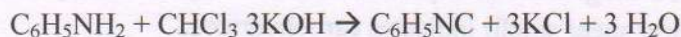
Note:- Do the following tests, only if N had been detected in Na fusion test.

7. TESTS FOR AMINES:

a. Solubility test: 0.1g sub. + 1ml. dil. HCl. Shake well. If the sub. Is soluble an gets reprecipitate an amine is indicated.



b. Carbylamines test: 0.11g. Sub. + 3dp. Chloroform + 2ml alc.KOH, shake well. Warm gently, offensive smell of isocyanides indicates primary amine (e.g., aniline)



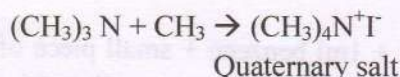
c. Dye test: Dissolve 0.1g. org. Compd. In 3ml. Dil.HCl. Cool (tap or ice) add 1ml. Sod. Nitrate (0.5g/H₂O) soln. And 1ml of a mixture of β -naphthol dissolved in NaOH. A scarlet red dye indicates a primary amine.

d. Acetylation: 0.1g.sub. + 1ml. Glacial HOAc + 0.5ml. acetic anhydride. Heat gently for 3-5mts., pour into 20 ml. Water in a beaker. White crystals indicate primary amine.



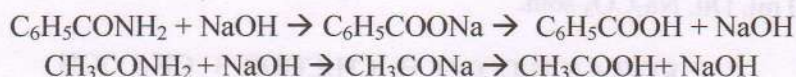
e. Liebermann's test: Dissolve 0.1g.sub. in 3ml. Ethyl alcohol, add 1ml. Con. HCl. Cool (tap or ice) add 1ml. NaNO₂ soln. Add 3ml ether, shake well an separate ether layer and evaporate it carefully. Add 5dp. Phenol to the residue. Warm, cool, and 5dp.Con. sulphuric acid. Pour the product into 20ml. Of dil.NaOH soln. First a red and then a blue soln. Indicates a secondary amine.

f. Quaternary salt test: 0.1g.sub. + 1ml. CH₃I soln. Shake well. Heat gently, allow it to stand for 2-5mts. White crystalline solid (quaternary salt) indicates tertiary amine.

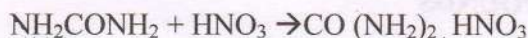


8. Tests for Amides:

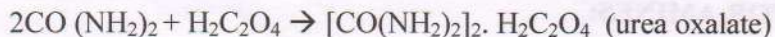
a. NaOH test: 0.1g. sub. + 2ml. 10% NaOH soln. Heat strongly. Smell of NH₃ indicates amide. Heat till no more NH₃ is evolved. Add 5dp. Con.HCl. white ppt. indicates aromatic amide, no ppt. Indicates aliphatic amide.



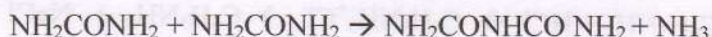
b. Urea nitrate test: 0.1g. Sub. + with 1ml.H₂O + Con. HNO₃. Shake well. White ppt. shows a dimide (like urea).



c. Urea-oxalate test: Shake 0.1g.sub. + 3ml. Satd.H₂C₂O₄ soln. White ppt. Shows a diamide

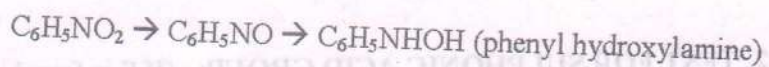


d. Biuret test: Heat 0.2g.sub. Strongly in a dry t.t. till no more Ammonia is evolved. Cool.add 1ml water an 5dp.CuSO₄ soln. And 5dp.NaOH soln. Violet colour shows diamide

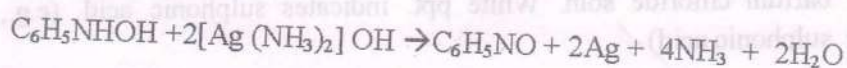


9. TEST FOR NITRO GROUP:

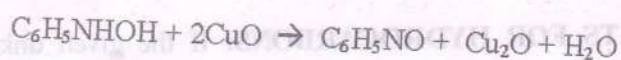
a. Mullikan and Barker's test: Dissolve 0.1g. sub. in a 2ml. Ethanol. Add 0.5g NH_4Cl and 0.5g. zinc dust. Heat for 2mts. Allow to stand for 3mts. Filter. Dissolve the filtrate into 2 parts



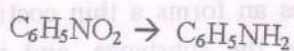
i.) To one part, add 2ml. Tollens reagent. Heat in water in water bath. Black ppt or Bright Ag mirror indicates Nitro group.



ii.) To other part, add 1ml each of Fehling's soln. "A" and "B". Heat for 2mts. A red ppt. (cuprous oxide) indicates Nitro group.

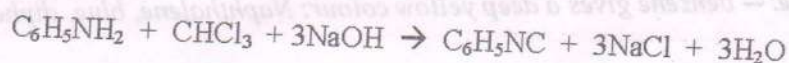
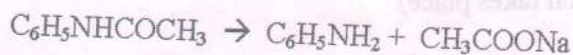


b. Reduction: To 0.3g. sub, add 5ml Con. HCl and piece of Sn, Heat for 3-5 mts. Filter. Ad KOH soln, till it become alkaline. An amine separates as a liquid (which may be tested by reaction under amines) it indicates Nitro compd.



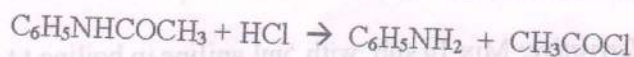
10. TESTS FOR ANILIDES:

a. Carbylamines test: 0.1g. sub. + 2ml. 10% NaOH soln. Heat for 2-3 mts. Add 2dp. chloroform. heat. Unpleasant smell (isocyanides) shows Anilide



b. Bromination: Dissolve 0.1g sub. Con. 3ml. glacial HOAc. Add 1ml bromine in HOAc. Shake well, pour into 10ml water. Pale yellow ppt. indicates Anilide.

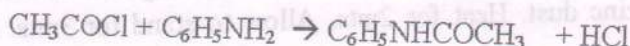
c. Dye test: 0.1g. sub. With 3ml. Con. HCl for 2-3mts. Cool the soln. (tap or ice). A slowly soln. Of 0.5g, sodium nitrite in water + soln of 0.5g β -naphthol in 2ml. NaOH. Scarlet red dye indicates anilide.



11. HALOGEN COMPOUNDS: (if Halogen is found in Na fusion test)

a. Alcoholic AgNO_3 test: 0.1g sub. + 2ml. Alc. AgNO_3 . Shake well. Gently warm. Formation of ppt. shows aliphatic halide or aromatic halide with halogen in aside chain (e.g. $\text{C}_6\text{H}_5\text{CH}_2\text{Cl}$) No ppt. aromatic compd. With halogen in nucleus.

b. Anilide test: 0.1g. Sub, + 1ml. Aniline. Shake well. Pour into 10ml water. White ppt. indicates acid chloride.



12. TEST FOR SULPHONIC ACID GROUP: (If S is found in Na fusion test)

a. Barium chloride test: Burn 0.1g. sub. in a Ni spatula. Dissolve residue in a dil. HCl, add barium chloride soln. White ppt. indicates sulphonic acid. (e.g., $\text{C}_6\text{H}_5\text{SO}_2\text{OH}$ - benzene sulphonic acid)

It is possible that the sulphonic acid forms, on decomposition, H_2SO_4 , which gives a white ppt. Of BaSO_4 with BaCl_2 .

13. TESTS FOR HYDROCARBONS: If the given unknown organic compd, does not answer for any of the above functional gps. It can be considered to be a hydrocarbon.

a. Test for aromatic hydrocarbons': dissolve 30mg. (or 3dps) of the substance in 1ml, of chloroform. In a small dry t.t, heat a small lump of fresh anhydrous AlCl_3 over tiny flame, until it slowly sublimes and forms a thin coating over the sides of the tube. Now add the chloroform solution of the substance, and roll the t.t., till the sublimate gets wetted completely.

An intense colour (yellow or greenish blue) develops, and this colour remains unchanged for a long time. This indicates the presence of an aromatic hydrocarbon. (Friedel-Crafts reaction takes place).

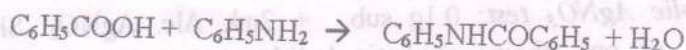
Note: — benzene gives a deep yellow colour: Naphthalene, blue, diphenyl, bluish- green.

F. DERIVATIVES

After identifying functional groups confirm by preparing a solid derivative as described below.

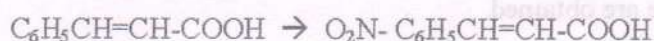
1. CARBOXYLIC ACID:

a. Anilide derivative: Mix 1g. sub. with 5ml aniline in boiling t.t. add porcelain bit, fit with air condenser and heat for 30 mts, in a sand bath. Pour hot mixture into 50ml, dil, and HCl stir well. Allow mixture to stand for 10-15min. White ppt. (anilide) separate out, filter, wash cold water, recrystallised from alcohol.



b. S-Benzyl isothiuronium derivative: Dissolve 1g. sub, in 10ml. 10% NaOH, and 10ml. Of 1g S-Benzyl thiuronium chloride in NaOH. Shake well. White ppt. obtained, filter wash with cool water.

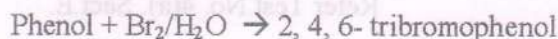
c. Nitro derivative: Unsatd. Acid (like cinnamic acid) can be nitrated using fuming nitric acid. Mix 1g sub. + 2ml fuming nitric acid slowly with constant stirring n awry t.t., keep aside for 10-15mts. pour into 50ml. Distilled water in a beaker. Yellow crystals separate out. Filter at pump, wash with water, recrystalised from alcohol.



d. Bromo derivative: For aromatic acid (salicylic acid and cinnamic acid) bromo derivative can be prepared. Mix 1g sub. With 5ml, Br_2 in glacial HOAc. With constant stirring, till yellow colour develops. Shake well for 10-15mts. pour it into 50ml water with stirring. Filter yellow crystals wash with water. Recrystalised from dil. Alcohol.

2. PHENOL:

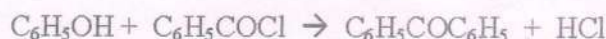
a. Bromo derivative:



b. Nitro derivative: Mix 1g sub. With 2ml Con. Sulphuric acid warm gently (2mts) cool, add 2ml Con. Sulphuric acid drop by drop, heat (10mts) pour into 20ml water. Yellow solid shows phenol group. Recrystalised small portion from boiling water.

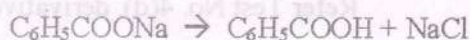
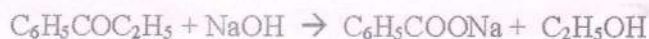


c. Benzyl derivative: 0.5g. sub. + 2ml. Water + 0.5ml. benzyl chloride + 5ml NaOH soln. Shake well. (5mts) pour into 20ml water (beaker). White ppt. (benzyl derivative of phenol).



3. ESTERS:

a. Acid derivative: To 0.5ml, sub. Add 10ml. 10% NaOH stir well for about 2-4mts. add Con. HCl. White ppt. shows an aromatic acid.



b. Amide derivative: Mix 0.5 ml sub. With 5ml. liq. NH_3 . Shake well, and leave for 10mts. the formation of white ppt. shows an amide.



4. ALDEHYDES AND KETONES:

a. *Semicarbozone derivative*: Mix 0.5ml. sub. with 0.5g. Semicarbazide hydrochloride and 0.5h. Sod. Acetate soln. Heat gently for about 10mts. and pour it into water. White crystals of semicarbazone are obtained.

Refer Test No. 4(c), Sect.E.

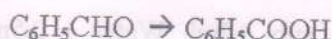
b. *Phenyl hydra zone derivative*: To 0.5ml. sub. mix 1ml. Phenyl hydrazine and 1ml. Glacial HOAc. Heat gently for about 10 mts. pours it into water. Yellow crystals show the derivative of carbonyl compds.

Refer Test No. 4(b), Sect.E.

c. *2,4-dinitrophenylhydrozone derivative*: To g.sub. Add 1ml. Borche's reagent and 1ml. Con.HCl. Heat for about 10mts. and pour it into water. Yellow crystals.

Refer Test No. 4(a), Sect.E.

d.) *Acid derivative*: For aromatic aldehydes, like benzaldehyde, acid derivative can easily be prepared by oxidation. Mix 1g. Sub. With 2ml 5% Na_2CO_3 soln. Add KMNO_4 soln. Drop by drop. Till the colour remains, heat for about 10mts. add dill. Sulphuric acid to the hot soln. Then a strong soln. of sod. Sulphite till the soln. Become colourless. On cooling, white crystals separate out.



5. CARBOHYDRATES:

Phenyl hydrazone derivative:

Refer Test No. 4(b), derivative.

6. ALCOHOL:

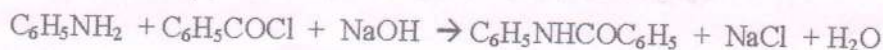
Acid derivative:

Refer Test No. 4(d), derivative.

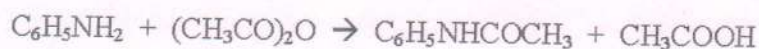
7. AMINES:

a.) *Benzyl derivative*:

Refer Test No. 4(c), derivative.

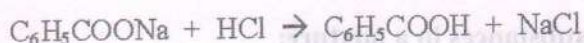
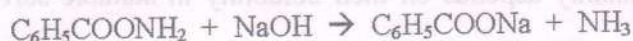


b.) *Acetyl derivative*: Mix g. Sub. With 1ml. Acetic anhydride and 1ml. Glacial acetic acid. Heat for abuit 10mts. pour into 20ml water. White crystals.



8. AMIDES:**Acid derivative:**

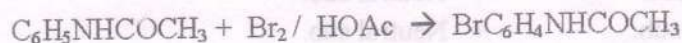
Refer Test No. 3(a), derivative.

**9. NITRO DERIVATIVE:**

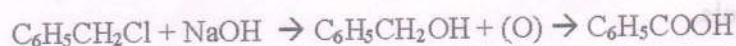
Reduce the nitro compd. To the corresponding amines by heating with tin and Hull.
convert the amine into acetyl or benzoyl derivative. Refer. 7(a) and 7(b),

10. ANILIDES:**Bromo derivative:**

Refer 1(d), derivative.

**11. HALOGEN COMPOUNDS:****a.) Acid derivative:**

Refer 3 (a) derivative.



b.) Nitro derivatives: Aromatic halogen compd. (with halogen in the nucleus) can be nitrated to get Nitro- derivative

Refer 2(b) derivative.

ANALYSIS OF MIXTURES

The identification of the components of a mixture of organic substances without separating them is difficult task. This is due to the presence of two or more functional groups, which may be function independent of each other. The separation of the components from a given mixture mainly depends on their solubility in suitable solvents, and their chemical properties.

Classification of substances in a mixture:

The components present in given mixture can be classified into the following four categories:-

- a. Acidic substances:
- b. Phenolic substances:
- c. Basic substances:
- d. Neutral substances.

The different combinations of organic substances that can be given in a mixture are listed below:-

- | | |
|-------------------|----------------|
| a.) Acid sub. | + Neutral sub. |
| b.) Basic sub. | + Neutral sub. |
| c.) Phenolic sub. | + Neutral sub. |
| d.) Acid sub | + Phenolic sub |
| e.) Phenolic sub. | + Basic sub. |

Acidic substances include carboxylic acids, sulphuric acids and rarely imides.

Phenolic substances are weakly acidic; they include phenol, substituted phenols and naphthols.

Basic substances include amino compounds and cyclic bases.

Neutral substances are generally classified into more reactive and less reactive substances. Alcohols, aldehydes, ketones, esters and carbohydrates contain more reactive functional groups. Among the less reactive compd, are the hydrocarbons and aromatic nitro compds.

PIOLET TESTS:

Before effecting the separation of the components from the bulk of the mixture, the method of separation must be determined. This is usually done subjecting a small amount of the following tests (called pilot tests):-

i). Soluble in water: About 0.2g. of the mixture is treated with 2ml. Of water. The mixture is shaken well, noting whether apart or the whole of the mixture has dissolved. The

clear solution is then tested with litmus, to find out whether the dissolved component is acidic. If component is soluble in water, its regeneration in a solid form is not possible.

ii.) **Solubility in ether:** About 0.2g. of the mixture is treated with 2ml of ether. The mixture is shaken well, nothing whether a part of the whole of the mixture has dissolved.

Three cases may be observed:

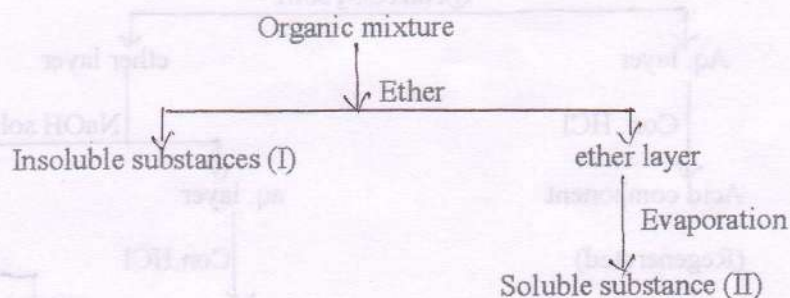
- Both components of the mixture are insoluble in ether.
- Only one component of the mixture is soluble in ether.
- Both components of the mixture are soluble in ether.

Case (a): Both components insoluble in ether

A mixture of this type is very rare. Only a very few mixtures can be made, where both components are insoluble in ether. Such a mixture combination is not recommended. The separation of the components from this mixture is beyond the scope of this lab. Manual.

Case (b): only one component soluble in ether

Separation of the components from a mixture of this type is easy. The insoluble component is filtered off, and the soluble component can be recovered by the evaporation of the ether layer. This method of separation is called *ether separation*.



Case(c): Both components soluble in ether

Organic mixtures of this type are common. Here, the components can be separated on the basis of their chemical behaviour. The mixture is treated with the following reagents in order: aq. Sodium bicarbonate, aq. NaOH and dill. HCl. The tests are carried out as follow:-

The organic mixture which has dissolved in ether is treated with 5ml. of aq. NaHCO_3 in a test tube. The mixture is shaken well, and then allowed to stand for 1 min. The two layers obtained are separated using a dropper. The bottom aq. layer is then treated with 2ml. of *Con. HCl*. The appearance of a precipitate indicates the presence of an aromatic acid. If no regeneration takes place. It indicates the absence of an acidic component in the mixture.

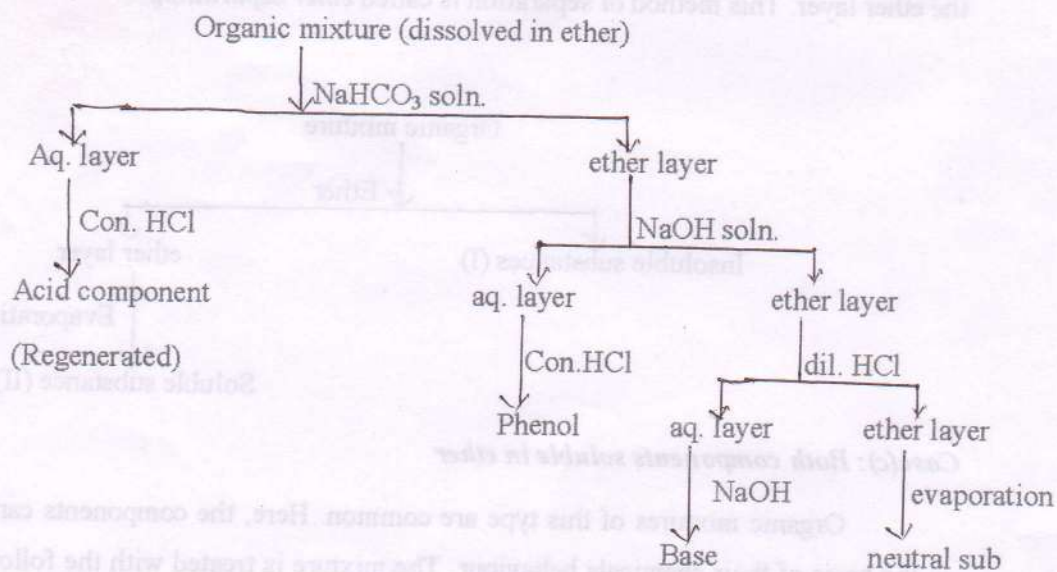
Note:- aliphatic acids (like oxalic acid and succinic acid) cannot be regenerated as solids. Due to their solubility in water. Hence, if no ppt appears after the neutralisation with Con.HCl, then about 10ml of ether is added to the aq.soln. and shaken well. The top ether layer is separated, and evaporated to dryness. An aliphatic acid will be obtained as a solid.)

The ether layer obtained from the above is then treated with 5ml. Of aq.NaOH. The mixture is shaken well, and allowed to stand for 1mts. the bottom aq. layer is separate, using a dropper, and tested with 2ml. Of con.HCl. A regeneration of a dissolved substance indicates the presence of a phenol. (A phenolic compound is considered as absent, if there is no regeneration on acidification of aq.layer.)

The ether layer obtained above is then treated with 3ml. Of dil. HCl. The mixture is shaken well, and allowed to stand for 1mts; the bottom aq. layer is separated using a dropper. And it is neutralised by adding 3ml. Of aq. NaOH. Regeneration indicates the presence of basic (amine) component. (The absence of regeneration indicates the absence of a basic component.)

A neutral substance, if present, is obtained by the evaporation to dryness of the ether layer (from above).

The separation process described above may be summarised, as follows:-

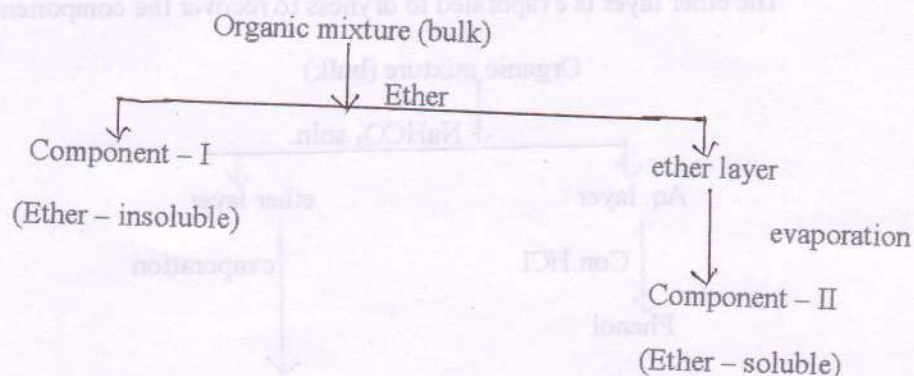


BULK SEPARATION:

After determining the separation procedure by doing the pilot tests outlined above, the bulk (major portion of the mixture) is taken, and the components are separated as follows:-

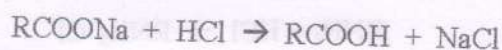
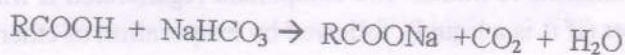
1.) **The separation** (only one component is soluble in ether).

About 4-5g. Of the given mixture (bulk) is shaken well with 10ml. Of ether. The clear ether layer is decanted. This procedure is repeated three times, to make sure that the ether-soluble component is completely dissolved (or extracted) from the mixture. This ether-soluble component is recovered by the evaporation to dryness of the combine ether layers.

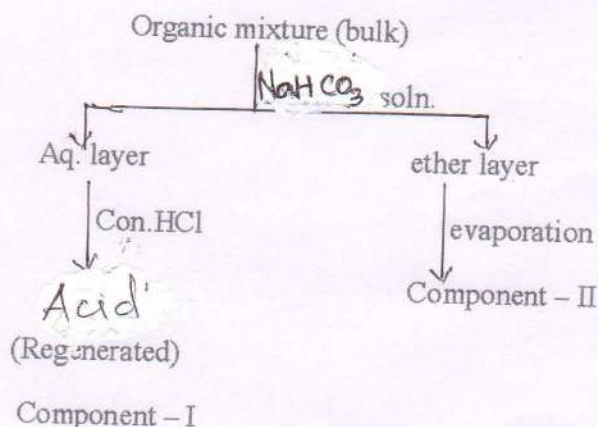


2. **Bicarbonate separation:** (mixture completely soluble in ether and one component is acidic.

About 4-5g. Of the organic mixture (bulk) is first dissolved in the minimum amount of ether. It is taken in a 250ml. Separating funnel, and 20ml. Of aq. NaHCO_3 soln. is added. The separating funnel is stoppered, shake well. And allowed to stand till two layers appear clearly separated. The bottom aq. layer is removed. Then, about 20ml. Of aq. NaHCO_3 is again added the separating funnel, and the procedure is repeated. The aq. layer is again collected, and combines with the first aq. layer already collected. The combined aq. layer are then acidified slowly by stirring with Con. HCl in drops, till the soln. Becomes acidic. The white precipitate regenerated is filtered off.

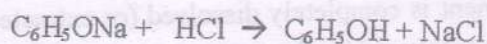
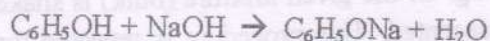


The ether layer is evaporated to dryness to recover the component - I

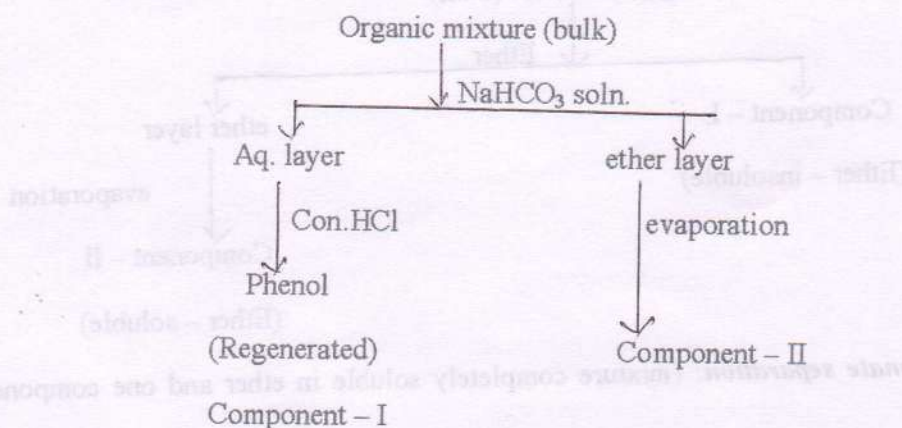


3. Alkali separation: (Mixture completely soluble in ether and one component is phenolic)

Follow the procedure given under bicarbonate separation, using aq. NaOH instead of aq. NaHCO₃.



The ether layer is evaporated to dryness to recover the component-II



4. Acid separation: (Mixture completely soluble in ether, and one component is basic).

About 4-5g. Of the organic mixture (bulk) is first dissolved in the minimum amount of ether. It is taken in a 250ml. Separating funnel, and 20ml. of dil. HCl is added. The separating funnel is stoppered, shaken well and allowed to stand for 5mts. the bottom aq. layer is removed. Then about 20ml of dil. HCl is added to the aq. layers are collected separately from the ether layer. The combined aq. layer are then neutralised slowly by stirring with aq. NaOH in drops, till the solution becomes alkaline to litmus. The component regenerated is filtered (if it is a solid), or extracted with ether (if it is a liquid), followed by evaporation of ether.

