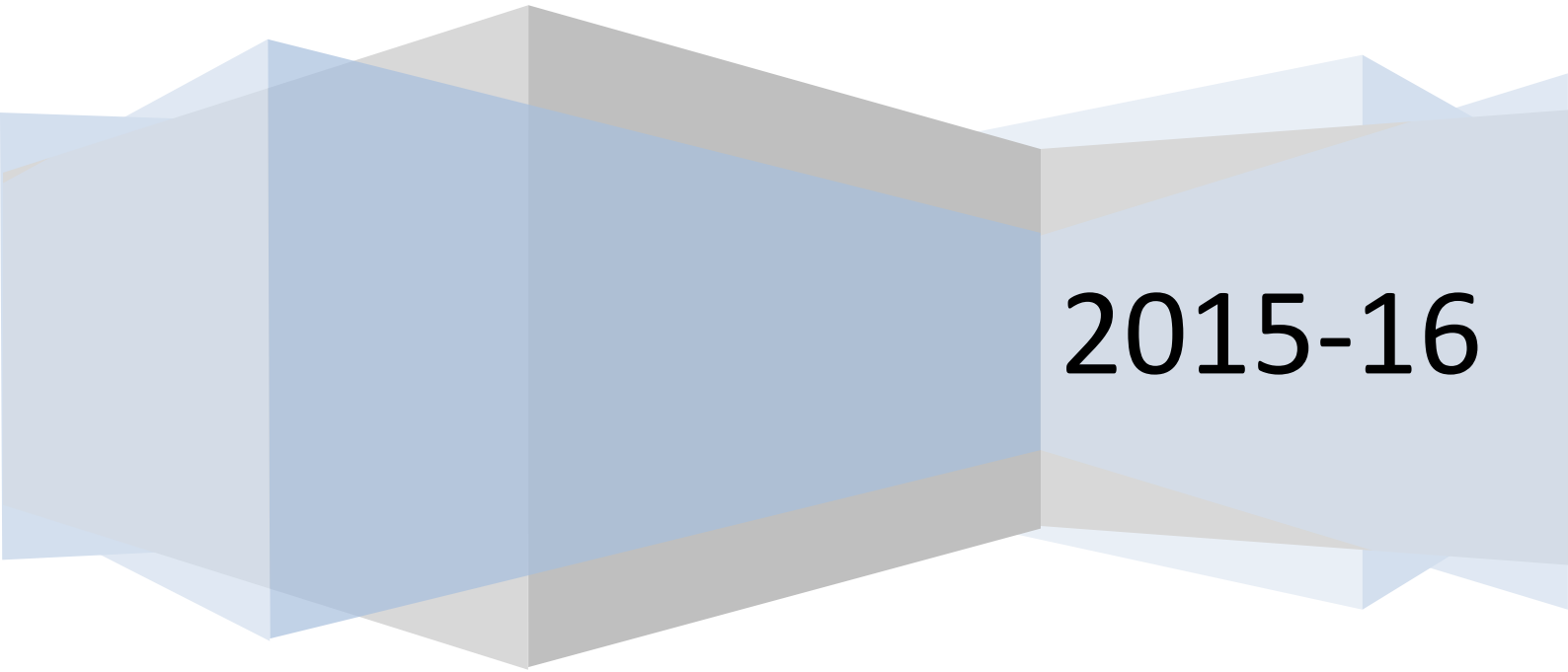
NORTH MAHARASHTRA UNIVERSITY, JALGAON

Standardized Practical Process Handbook

for

M.Sc.-Part-II(Organic Chemistry)



2015-16

1. Preparation of Benzanilide by Beckmann Rearrangement

Stage I: Benzene to Benzophenone

In a 100 ml round bottom flask place 5 ml of dry benzene and 8.75 gm of redistilled benzoyl chloride. Weigh out 9.25 gm of finally powdered anhydrous aluminum chloride in to a dry stoppered conical flask, and add the solid with frequent shaking, during 10 minutes to the content of the flask. Fit a reflux condenser with a glass absorption trap attachment to the flask, and heat on a water bath for three hours or until hydrogen chloride is no longer evolved, pour the content of the flask while still warm in to a mixture of 40-45 gm crushed ice and 25 ml concentrated hydrochloric acid. Separate the upper benzene layer (filter first, if necessary) wash it with 15 ml 5 percent aqueous sodium hydroxide solution, then with water and dry with magnesium sulphate. Remove the benzene after filtration by flash distillation and distilled the residue under diminished pressure through a short fractionating side arm. Collect the benzophenone at 187-190^{o}C/ 15mm Hg, it solidifies to a white solid on cooling. Record weight of the product, calculate the yield and its physical constant.

Reference- Vogel's Text book of practical Organic Chemistry, 5th edition

OR

In a dry 250 cc. flask put 5 grams of benzene, 5 grams of benzoyl chloride, 10 cc. of carbon bisulphide, and 6 grams of anhydrous aluminium chloride. Connect the flask with a reflux condenser and heat it on a water-bath for 2 hours. Cool the flask in water, and pour the contents into about 60 cc. of water and 4 cc. of concentrated hydrochloric acid. Separate the layer of carbon disulphide and evaporate off the solvent in a flask and distil off the solvent on a water-bath through a long condenser. Add 10 cc. of alcohol and a pinch of bone-black, boil for about 5 minutes, and filter hot. Wash the bone-black with hot alcohol. Cool and filter off the crystals by suction and wash them twice with a mixture, of 2 volumes of alcohol and 1 of water. Record weight of the product, calculate the yield and its physical constant.

Stage II: Benzophenone to Benzophenone oxime

Place a mixture of 3.12 gms of benzophenone, 2.5 gm. of hydroxylamine hydrochloride, 7 ml of rectified spirit and 1.2 ml of water in a 100 ml round bottom flask. Add 3.5 gm of sodium hydroxide (pellet form) in a portions with shaking. If the reaction becomes too vigorous, cool the flask under running tap-water. When all the sodium hydroxide has been added, heat to boiling and reflux for 5 minutes. Cool and pour the contents of the flask into 9.40 ml of conc. Hydrochloric acid in 60 ml of water contained in 250 ml beaker. Filter off the precipitate at the pump, Wash thoroughly with cold water and drain well and dry in oven at 40^{0}C or in vacuum designator. Recrystallise from about 20 ml of methyl alcohol. Record weight of the product, calculate the yield and its physical constant.

Reference- Vogel's Text book of practical Organic Chemistry, 5th edition

Stege- III: Benzphenone oxime to Benzanilide (Beckmann Rearrangement)

Dissolve 2 g of benzophenone oxime in 20 mL of anhydrous ether in a 100 ml round-bottomed flask and add 3 ml of pure thionyl chloride or 3 gm of powdered phosphorus penta chloride (this step should be carried out in a fume cupboard). Distill of the solvent or other volatile products on a water bath. Add 25 ml of water boil for several minutes and break up any lumps which may be formed. Decant the supernatant liquid (filter at the pump if necessary), and recrystallise the product from methanol (toxic!). Record weight of the product, calculate the yield and its physical constant.

Reference- Vogel's Text book of practical Organic Chemistry, 5th edition

2. Preparation of Anthranilic acid

Stage I - Phthalic acid to Phthalic Anhydride

Take 3 gm of phthalic acid in a porcelain dish which is gently heated on sand bath, the dish is covered with perforated filter paper over with inverted funnel is placed. Before place the funnel the cotton is injected in the funnel the vapour rising from it pass into the hole in filter paper before deposit solid on the wall of funnel. The outside cotton crystals are crude product and outside the recrystalised product. Record weight of the product, calculate the yield and its physical constant.

Stage II - Phthalic Anhydride to Phthalimide

Intimately mix 9.9 gm of Phthalic anhydride and 2.2 gm of urea and place the mixture in a 100 ml. long- necked R.B.flask. Heat the flask in an oil-bath at 130- 135^{O} C for about 15 minutes. When the contents have melted, effervences starts and the reaction mixture becomes solid. Remove the flame and allow to cool. Add about 8 ml. of water to disintegrate the solid in the flask, filter, wash the product with little water and dry at 100^{O} C. Determine the yield and m.p.

Stage III - Phthalimide to Anthranilic Acid

Prepare a solution of 6 gm. of NaOH in 24 ml. of water in a 100 ml. conical flask and cool to 0^{O} C. Add 5.12 gm. of bromine in one portion and stir until all the bromine has reacted. Cool the mixture to 0° C.

Meanwhile prepare a solution of 4.4 gm. of NaOH in 16 ml. of water. Add 4.8 gm of above product from stage I in one portion to the cold hypobromite solution. Stirr vigorously the contents of the flask and add the prepared NaOH solution rapidly. The solid will dissolve and the temperature will rise to about 70^{O} C. Warm the mixture to 80^{O} C for 2 min. Filter if necessary. Cool in ice and add conc. HCl slowly and with stirring until the solution is just neutral (about 12 ml. is required). Precipitate the product completely by addition of 4-5 ml. glacial acetic acid. Filter at the pump and wash with little water. Recrystallise with hot water. Record weight of the product, calculate the yield and its physical constant.

3. Preparation of Phthalimide

Stage I - Phthalic acid to Phthalic Anhydride

Take 3 gm of phthalic acid in a porcelain dish which is gently heated on sand bath, the dish is covered with perforated filter paper over with inverted funnel is placed. Before place the funnel the cotton is injected in the funnel the vapour rising from it pass into the hole in filter paper before deposit solid on the wall of funnel. The outside cotton crystals are crude product and outside the recrystalised product. Record weight of the product, calculate the yield and its physical constant.

Stage II - Phthalic Anhydride to Phthalimide

Intimately mix 9.9 gm of Phthalic anhydride and 2.2 gm of urea and place the mixture in a 100 ml. long- necked R.B.flask. Heat the flask in an oil-bath at 130- 135^{O} C for about 15 minutes. When the contents have melted, effervences starts and the reaction mixture becomes solid. Remove the flame and allow to cool. Add about 8 ml. of water to disintegrate the solid in the flask, filter, wash the product with little water and dry at 100^{O} C. Record weight of the product, calculate the yield and its physical constant.

4. Preparation of p-Aminobenzoic acid

Stage I: Toluene to p-Nitrotoluene

Add roughly 2 ml of toluene portion wise with shaking to 5ml of conc. HNO_3 at room temp. (cool under tap in case the reaction mixture tends to become warming).Stir the mixture for the 15 min and add some crushed ice when the product will be crystalline out either on standing or on scratching the sides of the test tube with a glass rod. Filter of the product. Record weight of the product, calculate the yield and its physical constant.

Stage II: p-Nitrotolune to p-Nitrobenzoic acid

Dissolve by stirring 2.5 gm of p-nitrotolune and 7.5 gm of sodium dichromate in a 15 ml water in 250 ml R.B flask. Add dropwise with stirring 10 ml of conc. sulphuric acid. During addition of sulphuric acid the temperature will rise and if the reaction tends to be vigorous reduce the rate of addition or cool the flask on tap water. After the addition is complete attach reflux condenser to the flask and heat on water bath for 30-40 minute. Cool and pour the reaction with stirring into a 200 ml beaker containing 60 ml ice water. Filter the precipitated acid through Buchner funnel and wash with cold water. Recrystallize from benzene and record weight of the product, calculate the yield and its physical constant.

Stage III: p-Nitrobenzoic acid to p-Aminobenzoic acid

In a 500 ml R.B flask place 2.5 gm of p-nitrobenzoic acid, 6 gm of powdered tin and 12.5 ml of conc.HCl. Attach the condenser to the flask and heat it over a low flam and when the reaction is commences, remove the heat source. Shake the flask gently for 20 minute so that all the tin dissolve and a clear solution is obtained. But if not then heat the flask again for 5 minute to complete the reaction. Cool the flask and filter. Add ammonium hydroxide solution to the filtrate until it is just alkaline, heat it on water bath for 25 minute and then filter. Wash the ppt with hot water. Combine the filtrate and washing and concentrate it to approximately 12 ml. acidify the conc. filtrate with glacial acetic acid and heat it on water bath until crystals start separating out. Cool the flask in ice bath and collect the crystals of p-aminobenzoic acid. Record weight of the product, calculate the yield and its physical constant.

5. Preparation of N-Bromo-succinimide

Stage I: Succinic acid to Succinic anhydride

In 250 ml round bottom flask, provided with the reflux condenser protected by the calcium chloride drying tube. Place 5.0 gm of Succinic acid and 8.65 gm of sulphuric acid **or** acetic anhydride, reflux the mixture gently on a water bath with occasional shaking until clear solution is obtained and then for the further hour to ensure the completeness of the reaction. Remove the complete assembly from the water bath and allow it to cool in ice. Collect the product on a Buchner funnel. Wash with two 20 ml portion of anhydrous ether and dry in a vaccum desiccator. Record weight of the product, calculate the yield and its physical constant.

Stage II: Succinic anhydride to Succinimide

Mix 2.5 gm of product of a **stage 1** and 0.56 gm of urea and place the mix in 100 ml long nacked R. B. flask. Heat the flask in a oil bath at 130 -135°C for about 15 min. when the content have melted effervence start and the reaction mix become solid. Remove the flame and allow to cool. Add about 2-3 ml of water to disintegrate the solid in the flask. Filter, wash with the product with little water and dry at 100°C. Record weight of the product, calculate the yield and m.p.

Stage III: Succinimide to N-Bromo-Succinimide

Take 1.5 gms of succinimide dissolve in a mixture of [0.6 gm sodium hydroxide, 5 gm crushed ice 15 ml water]. Cool the mixture in an ice bath and add 0.8 ml (2.47 gm) bromine at once while stirring fastly. Stir for five minutes, filter the precipitated product and wash with cold water (don’t wash too much). Dry the crude product using desiccator. Record weight of the product, calculate the yield and its physical constant.

6. Preparation of p-Chloro-nitrobenzene by Sandmeyer reaction

Stage- I: Acetanilide to p-Nitro acetanilide

In a 100 ml conical flask dissolve 3.5 gm of Acetanilide in 4 ml of glacial acetic acid by warming gently. Add slowly with swirling 5 ml ice-cold conc. H_2SO_4, the mixture becomes warm and clear solution results. Surround the beaker with a freezing mixture of ice and salt and stir mechanically. Prepare a nitrating mixture by adding 2 ml conc. HNO_3 to2.5 ml cold conc. H_2SO_4. Cool the solution to room temperature and transfer to a small separating funnel.

Cool the above acidic solution of the substance at 5°C in an ice-bath; run the nitrating mixture gradually while temperature of the mixture is maintained below 10^0c. After all the acid has been added, remove the beaker from the freezing mixture allow the solution to stand at room temp, for 1hour. Pour the solution slowly with stirring 20 gm of crushed ice, the crude p-nitro acetanilide is precipitated allow to stand for 15 minutes filter with suction on a Buchner funnel, wash with cold water until free from acid and drain well Recrystallise with ethanol. Record weight of the product, calculate the yield and its physical constant.

Stage-II: p-Nitro acetanilide to p-Nitroaniline

Boil the mixture of 3 gm of p-nitro acetanilide and 15 ml 70% sulphuric acid(12ml concentrated sulphuric acid cautiously addin thin stream with 9ml water)under reflux condenser for 20-30 minutes or until a test sample remains clear upon diluation with 2-3 times its volume of water. The p-nitroaniline is now present in the liquid as the sulphate. Pour the clear hot solution into 100 ml of cold water and precipitate the p-nitroaniline by adding excess 10% NaOH solution or concentrated ammonia solution.Cool the reaction mixture in ice water, if necessary. Filter the yellow crystalline precipitate at pump, wasgh it with water and drain thoroughly. Recrystallise it from a mixture of equal volumes of rectified spirit and water or from hot water. Filter wash and dry, record weight of the product, calculate the yield and its physical constant.

Stage-III: p-Nitroaniline to p-Chloro-nitrobenzene

Prepare a mixture of 10 ml conc. HCl and 10 ml H_2O by slowly adding the acid to the H_2O, and then dissolve 2.5 g of p-nitroaniline in it. Cool to 5^oC, and slowly add a solution of 3.5g $NaNO_2$ in 7.5ml water. Leave the solution to stand at 5^oC. Meanwhile prepare the reagent(CuCl) as follows.

Preparation of Reagent(CuCl) Add 7 g $CuSO_4.5H_2O$ and 2.35 g NaCl to 25 ml water in a 250mL beaker and warm the mixture to 55-60 ^{o} C, stirring with a glass rod until a clear solution is obtained. Add a solution of 3.5 g sodium bisulphite in 6.5ml water to the solution. The mixture will become green rapidly, and the CuCl that forms separates as a white powder as the solution. Cool the solution to 10-15^{o}C by standing in cold water to ensure the complete precipitation of the CuCl.

Allow the solution to stand in cold water. Filter off the solid CuCl on Buchner funnel under vacuum and wash with distilled water. Transfer the well-drained CuCl to a 250 ml beaker containing a mixture of 5 ml conc. HCl and 5 ml water mixed as above. A brownish-coloured solution will form. Heat the solution on a water bath.

Add the diazonium solution(**caution:** which must still below $5^{o}C$) prepared above into hot CuCl solution in small portions with vigorous stirring using a glass rod. When the addition is complete, allow the mixture to stand for 10 minutes, followed by heating on a water bath for 15 minutes. Cool, filter and wash the solid with distilled water. Recrystallise from methanol. Record weight of the product, calculate the yield and its physical constant.

7. Preparation of *p*-Iodo-nitrobenzene by Sandmeyer reaction

Stage- I: Acetanilide to p-Nitro acetanilide

In a 100 ml conical flask dissolve 3.5 gm of Acetanilide in 4 ml of glacial acetic acid by warming gently. Add slowly with swirling 5 ml ice-cold conc. H_2SO_4, the mixture becomes warm and clear solution results. Surround the beaker with a freezing mixture of ice and salt and stir mechanically. Prepare a nitrating mixture by adding 2 ml conc. HNO_3 to2.5 ml cold conc. H_2SO_4. Cool the solution to room temperature and transfer to a small separating funnel.

Cool the above acidic solution of the substance at 5°C in an ice-bath; run the nitrating mixture gradually while temperature of the mixture is maintained below $10^{0}c$. After all the acid has been added, remove the beaker from the freezing mixture allow the solution to stand at room temp, for 1hour. Pour the solution slowly with stirring 20 gm of crushed ice, the crude p-nitro acetanilide is precipitated allow to stand for 15 minutes filter with suction on a Buchner funnel, wash with cold water until free from acid and drain well Recrystallise with ethanol. Record weight of the product, calculate the yield and its physical constant.

Stage-II: p-Nitro acetanilide to p-Nitroaniline

Boil the mixture of 3 gm of p-nitro acetanilide and 15 ml 70% sulphuric acid(12ml concentrated sulphuric acid cautiously addin thin stream with 9ml water)under reflux condenser for 20-30 minutes or until a test sample remains clear upon diluation with 2-3 times its volume of water. The p-nitroaniline is now present in the liquid as the sulphate. Pour the clear hot solution into 100 ml of cold water and precipitate the p-nitroaniline by adding excess 10% NaOH solution or concentrated ammonia solution.Cool the reaction mixture in ice water, if necessary. Filter the yellow crystalline precipitate at pump, wash it with water and drain thoroughly. Recrystallise it from a mixture of equal volumes of rectified spirit and water or from hot water. Filter wash and dry, record weight of the product, calculate the yield and its physical constant.

Stage-III: p-Nitroaniline to p-Iodo-nitrobenzene

First prepare the diazonium salt from p-nitroaniline, for this mix 10 g ice and 10 ml water in a 250 ml beaker. Slowly add 2.0 ml concentrated sulfuric acid. Cool the solution to 5 °C in an ice bath. Slowly add 1.25 gm of p-nitroaniline in small portions using a piece of folded weighing paper as a funnel. Add a small portion with spatula and then stir. Most of the p-nitroaniline should dissolve.

Prepare a solution of 0.63 g sodium nitrite dissolved in 5 ml H_2O in a 100 ml flask. When the entire solid has dissolved, cool the solution to 5 °C in an ice bath and then slowly add the sodium nitrite solution to the solution of the p-nitroaniline at such a rate so as to maintain the temperature of the reaction below 10 °C. Keep the reaction mixture as cold as possible. Leave it in the ice bath at all times. Remember, you have just formed a diazonium salt and this is unstable if allowed to warm to room temperature. When you have added all of the sodium nitrite solution, add a spatula tip of urea and stir. (This will react with any excess nitroso ion.)

Prepare a solution of 2.5 gm of potassium iodide in 15 ml water in your 250 ml beaker. Slowly pour the diazonium salt solution into the potassium iodide solution with stirring. You will see lots of foaming at this point as the N_2 gas is released. Pour a little of the diazonium salt solution and then stir and wait for the foaming to subside. As you wait, return the diazonium salt solution to the ice bath so as to keep it as cold as possible. When all the diazonium salt has been added, wait for the foaming to subside and then cool the beaker in ice bath. Collect the solid product that has formed on the Buchner funnel using suction filtration. Remember to wet the filter paper before filtering. Recrystallise the product from isopropyl alcohol. Record weight of the product, calculate the yield and its physical constant.

8. Benzene to Benzophenone(Pinacol-Pinacolone Rearrangement)

Stage-I: Benzene to Benzophenone

In a 100 ml round bottom flask place 2 ml of dry benzene and 3.5 gm of redistilled benzoyl chloride. Weigh out 3.7 gm of finally powdered anhydrous aluminium chloride in to a dry stoppered conical flask, and add the solid with frequent shaking, during 10 minutes to the content of the flask. Fit a reflux condenser with a glass absorption trap attachment to the flask, and heat on a water bath for three hours or until hydrogen chloride is no longer evolved, pour the content of the flask while still warm in to a mixture of 20 gm crushed ice and 10 ml concentrated hydrochloric acid. Separate the upper benzene layer(filter first, if necessary) wash it with 5 ml 5 percent aqueous sodium hydroxide solution, then with water and dry with magnesium sulphate. Remove the benzene after filtration by flash distillation and distilled the residue under diminished pressure through a short fractionating side arm. Collect the benzophenone at 187-190^{o}C/15 mm Hg, it solidifies to a white solid on cooling. Record weight of the product, calculate the yield and its physical constant.

OR

OR

In a dry 500 cc. flask put 25 grams of benzene, 25 grams of benzoyl chloride, 50 cc. of carbon bisulphide, and 30 grams of anhydrous aluminium chloride. Connect the flask with a reflux condenser and heat it on a water-bath. for 2 hours. Cool the flask in water, and pour the contents into about 300 cc. of water and 20 cc. of concentrated hydrochloric acid. Separate the layer of carbon bisulphide and evaporate off the solvent in a flask and distil off the solvent on a water-bath through a long condenser. Add 50 cc. of alcohol and 2 grams of bone-black, boil for about 5 minutes, and filter hot. Wash the bone-black with hot alcohol. Cool and filter off the crystals by suction and wash them twice with a mixture, of 2 volumes of alcohol and 1 volume of water. Record weight of the product, calculate the yield and its physical constant.

Stage-II: Benzophenone to Benzpinacol

2.5 g of benzophenone was placed in a round bottom flask and dissolved in isopropanol (15ml) by warming on water bath. Add 4-5 drops of glacial acetic acid. The round bottom was stoppered and the reaction mixture was exposed to direct sunlight .Colourless crystals of product started appearing along the side of the round bottom flask after **5 hours**.The reaction mixture was allowed to stand in bright sunlight until no further solid appear to separate out (2-3 days).Cool the flask in ice bath. Filter the colourless needle like product. Record weight of the product, calculate the yield and its physical constant.

Stage-III: Benzpinacol to Benzpinacolone

2.0 g of the above product was placed in a round bottom flask and added a solution of iodine in glacial acetic acid (10 ml) and the reaction mixture was refluxed on a oil bath for 10-15 minutes. The reaction mixture was allowed to cool in ice bath. The crystals of product was filtered and dried. Record weight of the product, calculate the yield and its physical constant.

9. Preparation of Acetophenone (Fries rearrangement)

Stage-I Hydroquinone to Hydroquinonediacetate

Add one drop Conc. H_2SO_4 to a mix of 5.5 g of hydroquinone and 10.3 (9.6 ml) of A.R. acetic anhydride in a 50 ml conical flask. Swirl the contents of the flask gently, the mix warms up rapidly and the hydroqunione dissolve after 3-5 minutes pour the clear solution on to 40 gm of crushed ice. Stir, filter with the suction, and wash with 50 ml. of water. Recrystallize from 50% ethanol by volume; about 40 ml. volume is required. Record weight of the product, calculate the yield and its physical constant.

Stage-II Hydroquinonediacetate to 2,5-Dihydroxyacetophenone

Prepare a mixture of 4.0 g of dry hydroquinone diacetate and 8.7 g of anhydrous aluminium chloride in a glass mortar and introduce in 100 ml . round-bottom flask, fitted with a 12” air condenser protected by a calcium chloride tube and connected to a glass absorption trap immerse the

flask in an oil bath and heat slowly so that's the temp reaches 110-120°C at the end of about 30 minutes; the evolution of hydrogen chloride then being. Raise the temp slowly to 160-165°C and maintain the temp 1 hour remove the flask from the oil bath and allow to cool. Add 30 g. of crushed ice followed by 2 ml. of conc.HCl in the order to decompose the aluminium chloride. Filter the resulting solid with suction and wash it with two 10 ml portions of cold water. Recrystallize the crude product from 20 ml. of 95 % ethanol. Record weight of the product, calculate the yield and its physical constant.

10. Preparation of aromatic aldehyde by Vilsmeier-Haack reaction

Stage-I Aniline to Acetanilide (Green Chemistry Method)

A mixture of aniline / substituted anilines (3.3 g) and zinc dust (0.16 g) in acetic acid (10 ml) in 100 ml round bottom flask was heated over a gentle flame using water condenser. Heating was continued for about 45 min., the reaction mixture was then carefully poured in cold water (33 ml) in 250 ml beaker with vigorous stirring. The shining crystals of product were separated slowly. After 15 min, crystals were collected by filtration. The solid crystals were washed over the Buchner funnel with water and product was dried and crystallized in boiling water.

Stage-II Acetanilide to 2-Chloroquinoline-3-carbaldehyde (Vilsmeier-Haack reaction)

To a solution of acetanilide (5 mole) in dry DMF (15 mole) at 0-5^{0}C with stirring $POCl_3$ (60 mole) was added drop wise and the mixture stirred at 80-90^{0}C for time ranging between 2 hr. the mixture was poured into crushed ice, stirred for 5 min and the resulting solid filtered, washed well with water and dried. The compounds were purified by recrystallization from either ethyl acetate or acetonitrile.

11. Wittig Reaction

Stage-I : Anthracene to Anthraldehyde (Vilsmeier-Haack reaction)

To a solution of anthracene (5 mole) in dry DMF (15 mole) at 0-5^{0}C with stirring $POCl_3$ (60 mole) was added drop wise and the mixture stirred at 80-90^{0}C for time ranging between 2 hr. the mixture was poured into crushed ice, stirred for 5 min and the resulting solid filtered, washed well with water and dried. The compounds were purified by recrystallization from either ethyl acetate or acetonitrile.

Stage-II: 9-Anthraldehyde to Alkene (Wittig reaction)

Place smallest possible stirring bar in a large test-tube. Set the test-tube into a beaker or Erlenmeyer flask so that you can stand it on a stir-plate. Weigh out 0.300 g of 9-Anthraldehyde and add this to the test tube. Add two pipette of dichloromerhane(DCM) and stir well.

Weigh out 0.480 gm of the benzyltriphenylphosphonium chloride and place it into the test tube. Add one pipette of water using this to try to rinse down any phophonium salt that's stuck on the sides. Stirr the mixture vigorously, and then add 0.65 ml of 50 % sodium hydroxide solution by syringe.

Stirr the Solution vigorously for 10 minutes. Workup as - Dilute with 3 ml of dichloromethane and 5 ml of water, and pour the mixture into the separatory funnel.
Rinse the test tube with another 3 ml dichloromethane and 3 ml of water, and pour this also into the separatory funnel. Shake it up vigorously, and then allow time to settle. Pour the organic layer into the same 50 ml Erlenmaeyer flask. Add an additional 5 ml of dichloromethane to the separatory funnel, and shake vigorously again. Pour the organic layer into the same 50 ml Erlenmaeyer flask that has the other dichloromethane. Dry it with sodium sulphate. Filter the organic solution into a separate 50 ml Erlenmaeyer flask, using a funnel packed with glass wool to filter off the sodium sulphate. Rinse the original Erlenmaeyer and funnel with additional DCM. Add a boiling stick to your organic solution and then place the Erlenmaeyer in to a hot-water bath (250 ml beaker) to boil off DCM. Remove the Erlenmaeyer from the hot water bath.

Purify your alkene by recrystallizing from 1-propanol solvent. Rinse with very small amount(2 ml) of ice-cold propanol . Do not add water. Let things dry thoroughly before getting your yield and physical constant Weigh the product and calculate its % yield.

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