III SEMESTER

PRACTICAL MANUAL FOR SECOND B.Sc. CHEMISTRY

(w. e. f. 2020 – 2021)

PRACTICAL – 3(ORGANIC PREPARATIONS AND IR ANALYSIS)

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SYLLABUS

Course outcomes

On the completion of the course, the student will be able to do the following**:**

1. How to use glassware, equipment and chemicals and follow experimental procedures in the laboratory
2. How to calculate limiting reagent, theoretical yield, and percent yield
3. How to engage in safe laboratory practices by handling laboratory glassware, equipment, and chemical reagents appropriately
4. How to dispose of chemicals in a safe and responsible manner
5. How to perform common laboratory techniques including reflux distillation, recrystallization, vacuum filtration.
6. How to create and carry out work up and separation procedures
7. How to critically evaluate data collected to determine the identity, purity, and percent yield of products and to summarize findings in writing in a clear and concise manner

ORGANIC PREPARATIONS 40M

1. Acetylation of one of the following compounds:
   Amines (aniline, o-, m-, p- toluidine and o-, m-, p-anisidine) and phenols (β- naphthol, vanillin, Salicylic acid) by any one method

A) Using conventional method.

B) Using green approach

2. Benzoylation of one of the following amines
   (aniline, o-, m-, p- toluidine and o-, m-, p-anisidine)

3. Nitration of any one of the following:
   Acetanilide/nitrobenzene by conventional method

   Salicylic acid by green approach (using ceric ammonium nitrate)

IR SPECTRAL ANALYSIS 10M

IR Spectral Analysis of the following functional groups with examples

1. Hydroxyl groups
2. Carbonyl groups
3. Amino groups
4. Aromatic groups

SCHEME OF VALUATION

Record	5M
Viva	5M
Practical	40M

I. Organic preparations -- 30M	
Equation	5M
Procedure in 10 min	10M
M.P/B.P	5M
Reporting yield	10M
II. IR Spectral analysis -- 10M	
For each group data	10M

HOD OF CHEMISTRY

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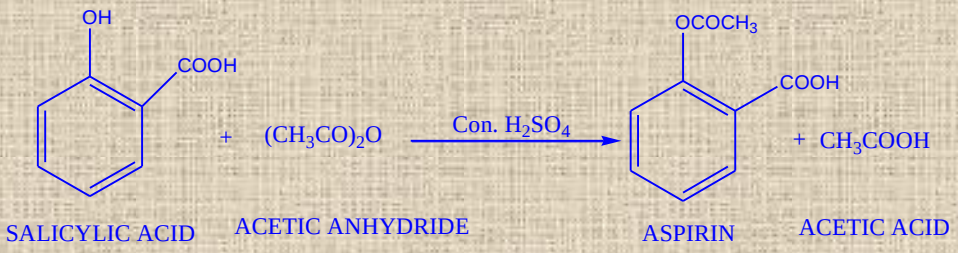
EXPERIMENT – 1

PREPARATION OF ASPIRIN

Principle

Aspirin is obtained by acylation of salicylic acid with acetic anhydride in the presence of Con. H_2SO_4.



SALICYLIC ACID ACETIC ANHYDRIDE ASPIRIN ACETIC ACID

Chemicals required

1. Salicylic acid – 1 g
2. Acetic anhydride – 3 to 4 mL
3. Con. H_2SO_4 – 0.2 mL

Apparatus

1. RB Flask
2. Dry boiling tube
3. Water bath
4. Glass rod
5. Filter paper

Procedure

Place a mixture of Salicylic acid of 1g and acidic anhydride about 3 to 4mL in dry boiling tube. Add the concentric H_2 SO_4 of 0.2 mL or 4 drops and shake the boiling tube. Now heat the reaction mixture at 50 to 60°C in water for 10 to 15 minutes stir continuously with a glass rod cool the reaction mixture and add water 10 to 15 mL. Filter the separated product and crystallised from dilute alcohol(50%.)

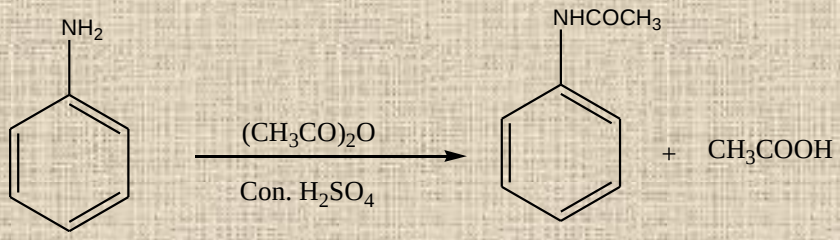
Report

EXPERIMENT – 2

ACYLATION OF ANILINE (AMINE)

Aim

To prepare acetanilide from aniline (aromatic primary amine) by acetylation.



ANILINE ACETANILIDE

Chemicals required

1. Aniline
2. Con.H2SO4
3. Acetic anhydride
4. Animal charcoal
5. Sodium acetate
6. Methylated spirit
7. Ice cold water

Apparatus required

1. 250 mL beaker
2. Measuring jar
3. Dropper
4. Whatmann filter paper
5. Suction pump
6. Funnel
7. Tripad stand
8. Wire gauge
9. Glass rod

Principle

Aromatic Amine undergo acylation in presence of acetic anhydride to form acylation product of aniline i.e acetanilide.

Clinical applications of acetanilide

Acetanilide used as analgesic as well as anti-pyretic like paracetamol

Procedure

Take 5mL of aniline in 250 mL Beaker and dissolve in 5mL of Con. H_2 SO_4 add about 100 mL of water. Add about 1 g of charcoal to the above mixture and warmed for about five minutes. Meanwhile dissolve 4 g of sodium acetate in about 15 mL water and keep in ice filter the above warmed aniline mixture with filter paper and add 6mL of acitic anhydride slowly with stirring until the compound is dissolved. This mixture into cooled sodium acetate solution, stirred vigorously. The formed acetanilide is filtered through suction pump and washed under dried outer a filtered paper and re-crystallised with 125mL boiling water containing methylated spirit.

Report

1. Yield – before purification = g
2. After re-crystallisation = g
3. M.P = ^{o}C

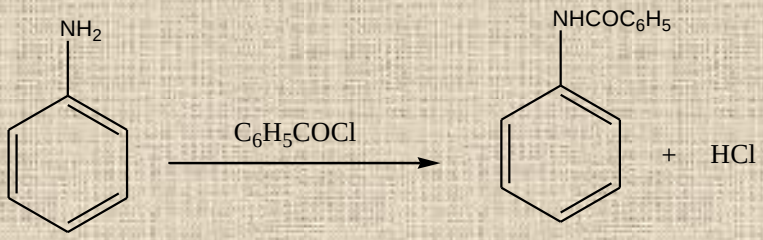
BENZYLATION OF AMINES
PREPARATION OF BENZONILIDE

Aim

To prepare benzonylide from aromaatic primary amine i.e. aniline.

Principle

Aromatic primary amines reacts with benzoylchloride undergoes electrophilic substituted to give benzonilide.



ANILINE BENZONILIDE

Chemicals required

1. Aniline
2. 10% NaOH
3. benzoyl chloride.

Apparatus

1. Iodine flask
2. 100 mL beaker
3. Glass rod
4. Measuring jar.

Procedure

Take 2.5 mL of aniline in an iodine flask add to20 mL of 10% NaOH solution dissolve this NaOH solution in aniline and add 3 mL of benzoyl chloride. The flask vigorously shaken for about 10-15 minutes. Heat is evolved in the reaction. So place the flask under water then the benzoyl derivation separated. A white powder when the reaction is completed when the odour of benzoyl chloride can be longer be detected. Water a cold mixture of about 5 mL of Con. Hno3 and about 3 mL of con. H2SO4 is prepared being this acid mixture into ice wate upto 5^0Cadd the above acetanilide solution drop by drop. After the addition of entire acid mixture this beaker is removed from ice and kept at room temperature from 15 minutes. Now this mixture is called into 100mL of ice water is separated and filtered. Through suction pump and now this residue is washed with cold water until it is free from acids the residue is from spirit and the crystals are dried near filter paper.

Report

1. Yield – before purification = g
2. After re-crystallisation = g
3. M.P = ^{o}C