V SEMESTER

PRACTICAL MANUAL FOR THIRD B.Sc. CHEMISTRY

(w. e. f. 2020 – 2021)

PRACTICAL – 7 (GREEN CHEMISTRY AND NANO TECHNOLOGY)

PREPARED BY

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SYLLABUS

LABORATORYCOURSE -7 30 Hrs (2 H / W)

PRACTICAL COURSE-7(GREEN CHEMISTRY AND NANO TECHNOLOGY) 50M
(At the end of Semester- V)

1. Identificationofvariousequipmentinthelaboratory.
2. Acetylationof1^{0}aminebygreenmethod: Preparationofacetanilide
3. Rearrangementreactioningreenconditions:Benzil-Benzilicacid rearrangement
4. Radicalcouplingreaction:Preparationof1,1-bis -2-naphthol
5. Greenoxidationreaction:Synthesisofadipicacid
6. Preparationand characterizationofbiodieselfromvegetableoil/ wastecookingoil
7. PreparationandcharacterizationofNanoparticlesofgoldusingtealeaves.
8. BenzoincondensationusingThiamineHydrochlorideasacatalystinsteadofcyanide.
9. Photo reductionofBenzophenonetoBenzopinacolinthepresenceofsunlight.

SCHEME OF VALUATION

Time:3 Hours Max Marks:50M

Record	5M
Viva voice	5M
Practical	40M

HOD OF CHEMISTRY

Head of the Dept. of Chemistry.
D.N.R. COLLEGE
BHIMAVARAM-534 202.

ACETYLATION OF 1^0AMINE BY GREEN METHOD

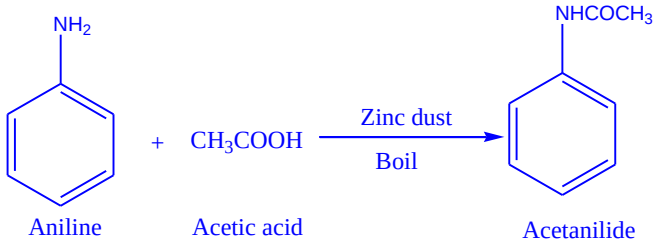
PREPARATION OF ACETANILIDE

Aim

To prepare acetanilide by acylation of 1^0 amine in green conditions.

Green method

Acetylation is carried out with acetic acid in the presence of zinc dust.



Chemicals required

1. Aniline – 10 mL (10.2 g)
2. Glacial acetic acid – 30 mL
3. Zinc dust – 0.5 g

Procedure

Add 10 mL of aniline and 0.5 g of zinc dust to 30 mL acetic acid in a 100 mL round bottom flask. Heat the mixture over a gentle flame using water condenser. Continue the heating for about 2 hrs. Pour the reaction mixture carefully in 100 mL cold water in a 250 mL beaker with cooling and vigorous stirring. Separation of shiny crystals of acetanilide from solution takes place. After 15 minutes, collect the acetanilide crystals by filtration. Wash the solid crystals over the Buchner funnel with water and dry the product.

Result

1. Yield of acetanilide…… g
2. Melting point of acetanilide is…….^{0}C

Mechanism

1. **Formation of zinc acetate**
2. **Attack of aniline on carbonyl caron of zinc acetate**
3. **Removal of acetate and zinc oxide**
4. **Deprotonation**

REARRANGEMENT REACTION IN GREEN CONDITIONS

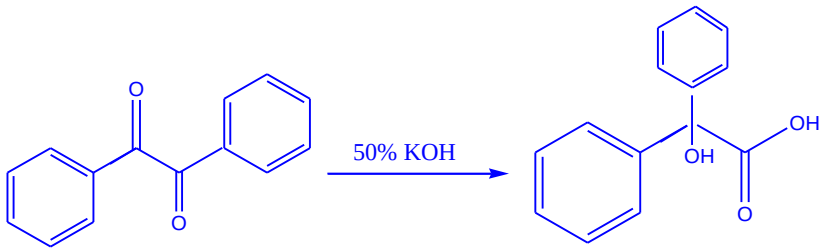
BENZIL – BENZILIC ACID REARRANGEMENT

Aim

To prepare benzilic acid from benzyl by benzyl-benzilic acid rearrangement in green conditions.

Green method

Preparation of benzilic acid in solid state under solvent free green condition.



Benzil Benzilic acid

Chemicals required

1. Benzyl – 1 g
2. Sodium hydroxide or potassium hydroxide – 1 g
3. Con. Hydrochloric acid

Procedure

Take 1 g of benzyl and 1 g of solid NaOH or KOH in a dry mortar. Grind the mixture thoroughly with the help of pestle to make an easy flowing powder. Take the powder in a dry conical flask and fit the mouth with a piece of cotton. Heat the conical flask on a boiling water-bath for 20 minutes. Then cool it to room temperature and dissolve the material in minimum amount of water. Remove unreacted benzyl, if any, simply by filtration. Acidify the aqueous solution with conc. HCl with thorough cooling in ice. Filter the precipitated benzilic acid, wash the filtrate with cold water and dry the product.

Report

1. Yield of benzilic acid….. g
2. Melting point of benzilic acid ……^{0}C.

Mechanism

1. **Attack of hydroxide anion on one of the carbonyl group of the benzyl (nucleophilic addition)**
2. **Migration of phenyl group with electrons to carbonyl carbon (1,2-shift)**
3. **Transfer of proton from carboxylic group to alkoxide group.**

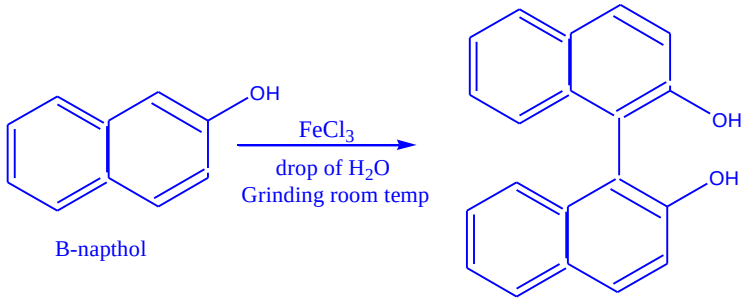
RADICAL COUPLING REACTION

PREPARATION OF 1, 1'-BIS-2-NAPHTHOL

Aim

To prepare 1,1'-bis-2-naphtol from β-naphthol by radical coupling reaction in green conditions.

Green procedure



B-napthol

1,1'-bis-2-naphthol

Chemicals required

1. B-naphthol – 2.88 g
2. Iron (III) chloride – 0.7 g
3. Water – 2 drops
4. Toluene (for recrystallization)

Procedure

Take 2.88 g of 2-naphthol and 0.7 g iron (III) chloride in an agate (or porcelain) mortar pestle and add 2 drops of water. Grind he mixture for 20 minutes. Allow the mixture to stand for about hours with a little grinding now and then. Transfer the mixture into a 100 mL beaker and add 40 mL of water. Boil the beaker for 10-15 minutes. Cool the mixture, filter the mixture, wash with 10 mL boiling water and dry the solid. Recrystallization the solid using toluene.

Report

1. Yield of 1,1'-bis-2-naphtol…….g
2. Melting point of 1,1'-bis-2-naphtol…. ^{0}C.

Mechanism

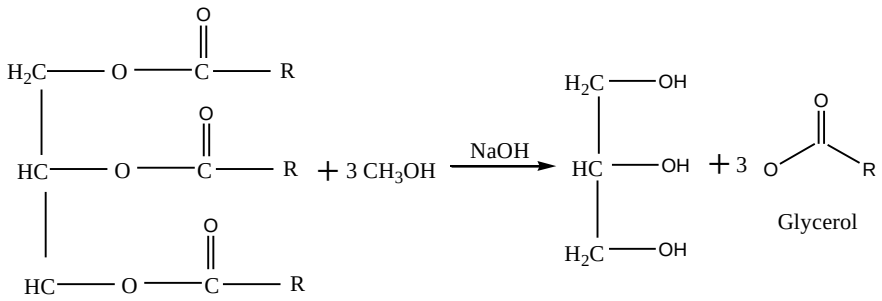
1. **Formation of free radical inpresence of metal oxidant.**
2. **Dimerization of free radicals (radical coupling reaction)**
3. **Rapid keto-enoltautomerism forms1,1'-bis-2-naphtol.**

PREPARATION AND CHARACTERISATION OF BIODIESEL FROM VEGETABLE OIL / WASTE COOKING OIL

Aim

To prepare biodiesel from vegetable oil by trans esterification reaction in green conditions.

Green procedure



Vegetable oil

Biodiesel

Chemicals required

1. Vegetable oil – 100 mL
2. Methanol – 20 mL
3. Sodium hydroxide – 3 pellets

Procedure

Add finely ground anhydrous sodium hydroxide into 20 mL of pure methanol (99% or higher purity) in a 250 mL Erlenmeyer flask. Stir the mixture vigorously until all the sodium hydroxide dissolved. Take 100 mL of pure vegetable oil in a 250 mL beaker and warm it about 40^0C. pour the warm oil into the methoxide solution in the flask with continuous stirring. At first the mixture would become cloudy but should soon two layers would separate stir the mixture for 15-20 minutes and transfer the contents of the flask into a 250 mL sparatory funnel. The mixture will separate into two different layers. The glycerol will fall to the bottom, and the methyl layer ester (biodiesel) will float to the top. Allow the experiment to sat for an hour. Open the stopcock of the separatory funnel and drain the glycerol into a small beaker.

Report

1. Yield of biodiesel…….g

Safety

1. **Methanol: flammable and poisonous. Dispose excess by allowing it to evaporate in the fume hood.**
2. **Sodium hydroxide: very corrosive. Causes severe burns. May cause permanent eye damage. Very harmful by ingestion.**

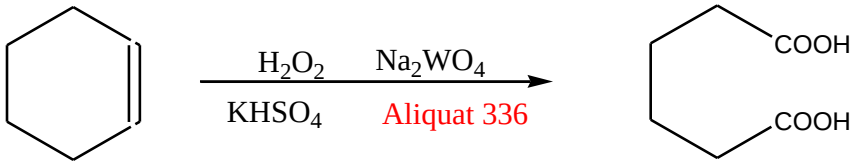
GREEN OXIDATION REACTION

SYNTHESIS OF ADIPIC ACID

Aim

To prepare adipic acid from cyclohexene in green conditions.

Green procedure



CYCLOHEXENE ADIPIC ACID

Chemicals required

1. Cyclohexene – 2 g
2. Sodium tungstate – 0.5 g
3. Potassium hydrogen sulphate – 0.37 g
4. Aliquat 336 – 0.5 g
5. Hydrogen peroxide (30%) – 12 mL

Procedure

Take 0.50 g of sodium tungstate dehydrate in a 50 mL round bottom flask fitted with a condenser. Add 0.5 g of aliquat 336, 12 mL of 30% hydrogen peroxide and 0.37 g of potassium hydrogen sulphate. Shake the mixture vigorously and add 2 g of cyclohexene. Heat the reaction mixture on a sand bath to reflux for 2 hrs. After two hours of reflux, remove the round-bottom flask from the sand bath. Upon cooling, the crude adipic acid is precipitated. Recrystallize the crude sample from water to get pure adipic acid.

The progress of the reaction was monitored by observing whether the layers are separated. As the liquid cyclohexene was converted to the water soluble adipic acid, the organic layer will eventually disappear.

Report

1. Yield of adipic acid….. g
2. Melting point of adipic acid…….^{0}C