ORGANIC CHEMISTRY

PRACTICAL-II

M.Sc. CHEMISTRY

SEMESTER-I, PAPER-VI

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M.Sc. CHEMISTRY: ORGANIC CHEMISTRY

PRACTICAL-II

First Edition : 2025

No. of Copies :

This book is exclusively prepared for the use of students of M.Sc. Chemistry, Centre for Distance Education, Acharya Nagarjuna University and this book is meant for limited circulation only.

Published by:

Prof. V. VENKATESWARLU
Director, I/c
Centre for Distance Education,
Acharya Nagarjuna University

Printed at:

FOREWORD

Since its establishment in 1976, Acharya Nagarjuna University has been forging ahead in the path of progress and dynamism, offering a variety of courses and research contributions. I am extremely happy that by gaining 'A+' grade from the NAAC in the year 2024, Acharya Nagarjuna University is offering educational opportunities at the UG, PG levels apart from research degrees to students from over 221 affiliated colleges spread over the two districts of Guntur and Prakasam.

The University has also started the Centre for Distance Education in 2003-04 with the aim of taking higher education to the door step of all the sectors of the society. The centre will be a great help to those who cannot join in colleges, those who cannot afford the exorbitant fees as regular students, and even to housewives desirous of pursuing higher studies. Acharya Nagarjuna University has started offering B.Sc., B.A., B.B.A., and B.Com courses at the Degree level and M.A., M.Com., M.Sc., M.B.A., and L.L.M., courses at the PG level from the academic year 2003-2004 onwards.

To facilitate easier understanding by students studying through the distance mode, these self-instruction materials have been prepared by eminent and experienced teachers. The lessons have been drafted with great care and expertise in the stipulated time by these teachers. Constructive ideas and scholarly suggestions are welcome from students and teachers involved respectively. Such ideas will be incorporated for the greater efficacy of this distance mode of education. For clarification of doubts and feedback, weekly classes and contact classes will be arranged at the UG and PG levels respectively.

It is my aim that students getting higher education through the Centre for Distance Education should improve their qualification, have better employment opportunities and in turn be part of country's progress. It is my fond desire that in the years to come, the Centre for Distance Education will go from strength to strength in the form of new courses and by catering to larger number of people. My congratulations to all the Directors, Academic Coordinators, Editors and Lesson-writers of the Centre who have helped in these endeavors.

Prof. K. Gangadhara Rao
M.Tech., Ph.D.,
Vice-Chancellor I/c
Acharya Nagarjuna University.

M.Sc. CHEMISTRY
SEMESTER-I, PAPER-VI
106CH24 - ORGANIC CHEMISTRY
PRATICAL-II

SYLLABUS

LIST OF EXPERIMENTS:

1) One step & two step Organic compounds preparation - Yield of crude and crystallized samples and reporting of the melting point/Boiling points.

   Preparations:

   i) Iodoform ii) n-Dinitroderivative iii) Asprin iv) p-Nitroaniline

   v) Bezophenone vi) Benzoic acid v ii) p-Bromo Acetanilide

   viii) Acetanilide ix) any other organic compound

2) Purification of organic compound-The student has to do Recryastallization to final compound(s) (for both steps) and submit the sample.
3) Distillation of Alcohol, Toluene.
4) Chromatography- The student has to submit purity of the final product with TLC
5) Chromatographic separation of impurities by TLC.
6) Student should practice solvent extraction methods.

Note: Apart from (1) & (2) each student must practice S.No. (3) to (6).

ACHARYA NAGARJUNA UNIVERSITY: CENTRE FOR DISTANCE EDUCATION

M.Sc. – Chemistry - Program code: 04

Program Structure

Program code	Program	Internal assessment	External exams	Max. Marks	credits
SEMISTER 1					
101CH24	Inorganic Chemistry-I	30	70	100	4
102CH24	Organic Chemistry-I	30	70	100	4
103CH24	Foundation for Chemistry	30	70	100	4
104CH24	Physical Chemistry-I	30	70	100	4
105CH24	Inorganic & Physical Chemistry **Practical-I**	30	70	100	4
106CH 24	Organic Chemistry **Practical-II**	30	70	100	4
SEMISTER 2					
201CH24	Physical Chemistry-II	30	70	100	4
202CH24	Organic Chemistry-II	30	70	100	4
203CH24	Essential Lab Techniques for Industry	30	70	100	4
204CH24	Inorganic Chemistry-II	30	70	100	4
205CH24	Inorganic & Physical Chemistry **Practical-I**	30	70	100	4
206CH24	Organic Chemistry **Practical-II**	30	70	100	4
SEMISTER 3					
301CH24	Applied Inorganic Analysis	30	70	100	4
302CH24	Analysis of Applied Industrial Products	30	70	100	4
303CH24	Optical Thermal & Radiochemical Methods of Analysis	30	70	100	4
304CH24	Principles and Techniques in Classical Analysis	30	70	100	4
305CH24	Classical Methods of Analysis **Practical-I**	30	70	100	4
306CH24	Instrumental Methods of Analysis **Practical-II**	30	70	100	4
SEMISTER 4					
401CH24	Advanced Methods of Analysis	30	70	100	4
402CH24	Analysis of Drugs, Foods, Diary Products & Biochemical Analysis	30	70	100	4
403CH24	Separation Techniques & Electro Analytical Techniques	30	70	100	4
404CH24	Environmental Chemistry & Analysis	30	70	100	4
405CH24	Classical & Instrumental Methods of Analysis **Practical-I**	30	70	100	4
406CH24	Spectral Problems **Practical-II**	30	70	100	4

CONTENTS

M.Sc. DEGREE EXAMINATION, MODEL QUESTION PAPER

M.Sc. CHEMISTRY - FIRST SEMESTER

ORGANIC CHEMISTRY (106CH24)

PRACTICAL - II

Max. Marks: 100 (Internal-30M & External-70M)

LIST OF EXPERIMENTS:

1) One step & two step Organic compounds preparation - Yield of crude and crystallized samples and reporting of the melting point/Boiling points.

   Preparations:

   i) Iodoform ii) n-Dinitroderivative iii) Asprin iv) p-Nitroaniline

   v) Bezophenone vi) Benzoic acid v ii) p-Bromo Acetanilide

   viii) Acetanilide ix) any other organic compound

2) Purification of organic compound-The student has to do Recryastallization to final compound(s) (for both steps) and submit the sample.
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4) Chromatography- The student has to submit purity of the final product with TLC
5) Chromatographic separation of impurities by TLC.
6) Student should practice solvent extraction methods.

Note: Apart from (1) & (2) each student must practice S.No. (3) to (6).

EXPERIMENT - 1

PREPARATION OF IODOFORM

AIM: To Prepare pure sample of Iodoform from acetone

CHEMICALS REQUIRED:

Iodine	– 5 g
5% Sodium Hydroxide	– 10 ml
Potassium Iodide	– 5 ml
Acetone	– 3 ml

PROCEDURE:

Dissolve 5 g of iodine in 5 ml acetone in a 100 ml conical flask or round bottomed flask. Add 5% NaOH solution in small portions with constant shaking the flask. Cool the flask from time to time under tap water so that temperature does not rise above 40°c. The addition of NaOH solution is further continued till the brown colour of iodine just disappears then, allow the flask to stand at room temperature for 5-10 minutes. Filter the iodoform, wash with little cold water and then dry on a filter paper. Recrystallize the crude iodoform by addition of small amount of rectified spirit in a 100 ml conical flask and heat it on a water bath. Finally, Filter the crystals of Iodoform and dried the crystals.

REPORT:

The yield of Iodoform is_____g.

The melting point of Iodoform is_____°C

$$CH_3COCH_3 + 3NaOI \rightarrow CH_3COCI_3 + 3NaOH$$

$$CH_3COCI_3 + NaOH \rightarrow CHI_3 + CH_3COONa$$

EXPERIMENT - 2

PREPARATION OF META - DI NITRO BENZENE

AIM: To prepare m - Di-Nitro Benzene from Nitro Benzene by Nitration.

CHEMICALS REQUIRED:

Nitro Benzene - 3 ml

Conc.HNO3 - 3.5 ml

Conc. H2SO4 - 5 ml

PROCEDURE:

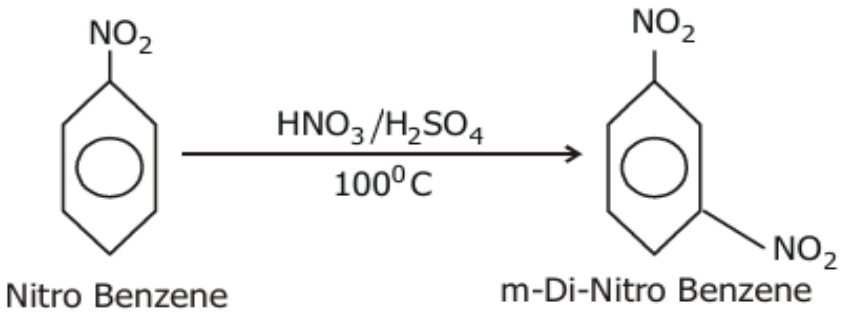
In 100 ml of round-bottomed flask, 3.5 ml of Conc.HNO3 & 5 ml of Conc. H2SO4 are taken. 3ml of Nitro Benzene is added in small portions and shaken well. For each addition, reflux the addition on a water bath for about 30 minutes with a continuous shaking.

Transfer the reaction mixture into 250 ml of crushed ice. Filter, washed with cold water and dried. Re-crystallize the crude M-di Nitro Benzene from Ethanol.

REPORT:

The yield of M- Di-Nitro Benzene is…….g.

The melting point of M- Di-Nitro Benzene is……..°C.



EXPERIMENT - 3

PREPARATION OF ASPIRIN

AIM: To Prepare Aspirin from Salicylic Acid.

CHEMICALS REQUIRED:

Salicylic Acid - 5 g

Acetic Anhydride - 7.5 ml

3M NaOH - 25 ml

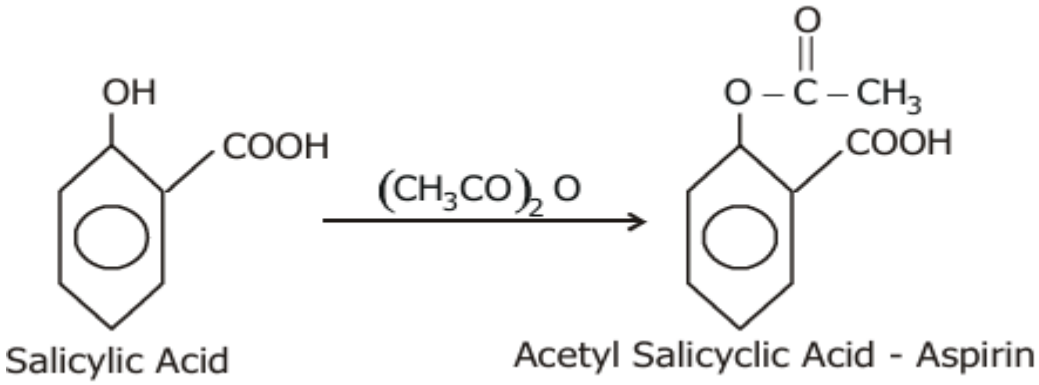
PROCEDURE:

Dissolve 5gms of Salicylic Acid in 25 ml of 3 M NaOH solution. Add 15 to 20 gms of crushed ice followed by Acetic Anhydride. Shake the mixture for about 30 to 60 seconds. The Aspirin separates in a practically pure condition either at once or after acidifying or by the addition of mineral acid (Conc. HCl). Collect the compound. Filter and Re-crystallize it from hot water.

REPORT:

The yield of Aspirin is ……g. .

The melting point of aspirin is ……°C



EXPERIMENT - 4

PREPARATION OF BENZOPHENONE

AIM: To Prepare Benzophenone from benzene

CHEMICALS REQUIRED:

Benzene	– 5 ml
Benzoyl Chloride	– 8.75ml
Aluminium trichloride	– 9.25 g

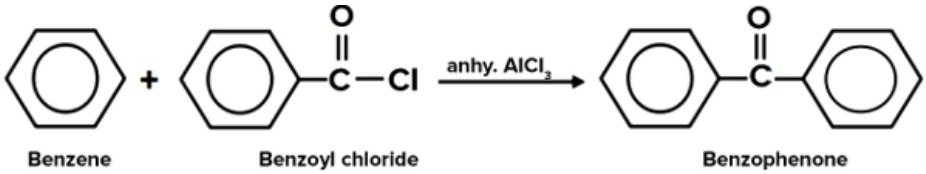
PROCEDURE:

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 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 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 187-190oC/ 15mm Hg, it solidifies to a white solid on cooling. Record weight of the product, calculate the yield and its physical constant.

REPORT:

The yield of Benzophenone is…….g. .

The melting point of Benzophenone is…….°C



EXPERIMENT - 5

PREPARATION OF P - NITROANILINE

AIM: To Prepare P – Nitroaniline from P – Nitroacetanilide

CHEMICALS REQUIRED:

P-nitro acetanilide – 5 g

Sulphuric acid – 10 ml

10 % NaOH – 10 ml

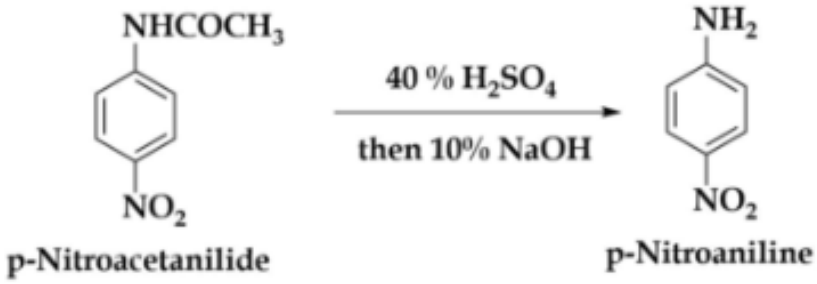
PROCEDURE:

Heat a mixture of 5.0 g of p-nitro acetanilide and 30 mL of 70% sulphuric acid in a 100 mL round bottomed flask fitted with reflux condenser for 20 minutes until a test sample remains clear upon dilution 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 in to 250 mL cold water and precipitate the p-nitroaniline by adding of 10 % NaOH solution filter of the yellow crystalline ppt. wash with water recrystallize from hot water m. p. = 148 °C

REPORT:

The yield of P – Nitroaniline is…… g

The melting point of P – Nitroaniline is……..°C



p-Nitroacetanilide p-Nitroaniline

EXPERIMENT - 6

PREPARATION OF BENZOIC ACID

AIM: To prepare Benzoic acid by the Oxidation of Benzyl Chloride.

CHEMICALS REQUIRED:

Benzyl Chloride	- 3 ml
Potassium Permanganate	- 7 g
Sodium Carbonate (Na2CO3)	- 1 g

PROCEDURE:

Suspend in a 250 ml round bottomed flask, 3ml of Benzyl chloride in 125ml of boiling water to which about 1 g of Sodium Carbonate is added. Introduce slowly 7 g of finely powdered KMnO4.

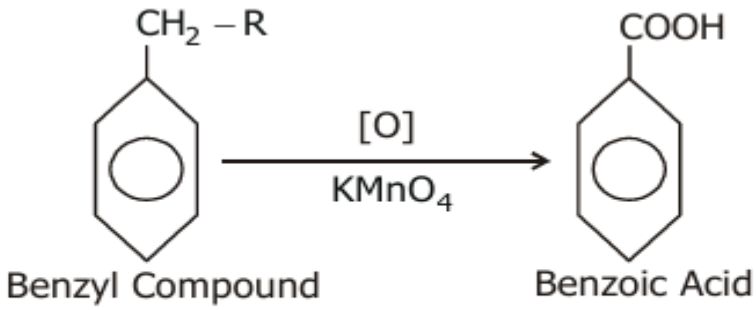
Heat under reflux until the purple color of KMnO4 disappears (1to4 hours).

Allow the mixture to cool and filter. Carefully acidify the filtrate with Conc. HCl. Filter the ppt of Benzoic acid and re-crystallize it from hot water.

REPORT:

The yield of Benzoic acid is……g. .

The melting point of Benzoic acid is……..°C



Where R = –H, – CH_3, – X, – NH_2

EXPERIMENT - 7

PREPARATION OF P-BROMO ACETANILIDE

AIM: To prepare p-Bromo Acetanilide from Acetanilide.

CHEMICALS REQUIRED:

Acetanilide - 13.5 g

Glacial Acetic Acid - 45 ml

Bromine - 5.3 ml

PROCEDURE:

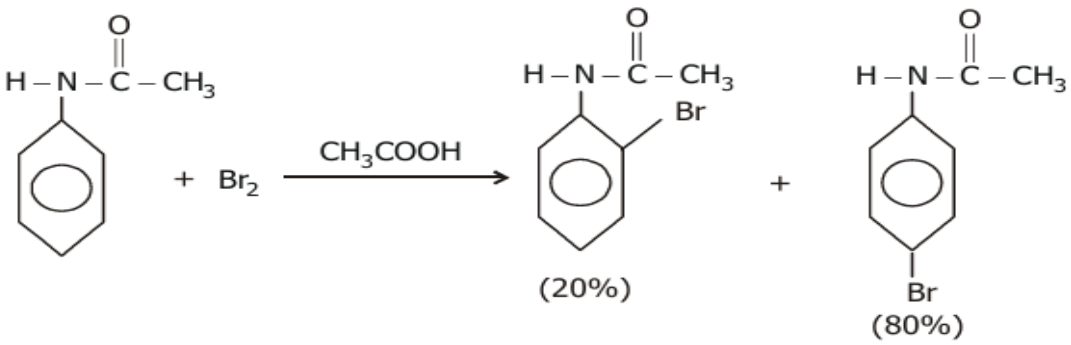
Dissolve 13.5 g of Acetanilide in 25 ml of Glacial acetic acid in 250 ml Conical flask. To this add 30 ml of Bromine solution (5.3 ml of Bromine in 25 ml of Glacial Acetic acid) drop by drop from Separating funnel.

The process is to be continued for half an hour. The conical flask is to be shaken well till entire process is completed. During the process of addition, the flask should be placed in a water bath containing ice-cold water. At one instance, a yellow precipitate is observed in the conical flask. Stop the process and transfer the entire mixture in the conical flask into the beaker, which is filled with ice blocks (or) Ice-cold water. Filter the precipitate and see that no moisture is present and collect the entire mixture and stored it.Take a small amount of the mixture and re crystallize with alcohol.

REPORT:

The yield of p- Bromo Acetanilide is…….g. .

The Melting point of p- Bromo Acetanilide is …….^{o}C



EXPERIMENT - 8

PREPARATION OF ACETANILIDE

AIM: Prepare Acetanilide from Aniline.

CHEMICALS REQUIRED:

Aniline – 10ml

Acetic Anhydride – 10ml

Glacial Acetic Acid – 10ml

PROCEDURE:

Take 10ml of Aniline into a 250ml round-bottomed flask equipped with reflux condenser. To this 10ml of acetic acid and 10ml of acetic anhydride are added. Then the mixture is refluxed for 30 to 60 minutes. Transfer the contents of the flask into 250 ml ice-cold water. Filter the crude at the Pump. Yellow coloured precipitate is formed on the filter paper.

Re-Crystallize the crude Acetanilide from Acetic Acid or from hot water. The crude product is boiled in water and saturated solution is prepared. The suspended impurities are remembered by filtering the hot solution. After cooling the crystals of acetanilide separate.

REPORT:

The yield of Acetanilide is……g.

The melting point of Acetanilide is……°C