Solvent free Cannizzaro reaction applying grindstone technique

A Dissertation report to the Department of Chemistry Sonari College, Sonari

Affiliated to Dibrugarh University

SUBMITTED BY -:

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Session -: 2024

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CERTIFICATE

This is to certify that the dissertation report entitled,**Solvent Free "Cannizzaro reaction by grind stone technique "**submitted to the Department of Chemistry, Sonari College is arecord of project work carried out by Lovita Teli.

The project meets all the requirements of B.Sc. project.

Further, it is also certified that the project has not been submitted for any purposes elsewhere.

Place: Sonari College

(Amrit Kumar Borpuzari)

t of Chemistry

e, Sonari

-785690, Assam, India

Dr Bikash Kumar Sarmah

Assistant Professor, Dept. of ChemistryDate:

CERTIFICATE

This is to certify that the project work entitled **Solvent Free "Cannizzaro reaction by grind stone technique"** Submitted by Lovita Teli a student of 6th semester to the Department of Chemistry, Sonari College, Sonari, affiliated to Dibrugarh University is carried out under my supervision and is found suitable to submit for the partial fulfillment of the B.Sc. Degree in Chemistry. This work, in the present form or in part, has not been submitted for any purpose elsewhere.

I wish her great success in future.

Signature of Guide

Place: Sonari College   (Dr. Bikash Sarmah)

ACKNOWLEDGEMENT

At the very outset, I would like to offer my sincere thanks and gratitude to Dibrugarh University for including a project work in the BSc. Semester syllabus

I would like to express my bumble gratitude to Dr. Bikash Kumar Sarmah Assistant professor of the Dept. of Chemistry Sonari College for his valuable guidance and encouragement to complete my project in a stipulated frame

I would like to extend my sincere thanks to Mr Suchitra Narayan Rajkhuwa and Mr. Amrit Kumar Borpuzari for extending their helping hand and support.

I would also like to thanks my friends and family member for their suggestion and support though my project work,I attained considerable practical knowledge and some other valuable ign too. This would not have been possible without their cooperation and contribution.

Date: 22/05/24

Place: Sonari College

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CONTENTS

Abstract :

Cannizzaro reaction of various aryl aldehydes employing solvent free grindstone technique using solid sodium hydroxide was examined and obtained excellent yields (94–99%) with 97–100% conversion rate. this reaction is very simple and has many benefits including cheap and easily available compound, good yield of product, simple experimental condition and without generation of pollution in shorter reaction time. The synthesized product was identified by itsphysical properties, melting point, TLC and characterized by UV Spectroscopy and NMR spectroscopy.
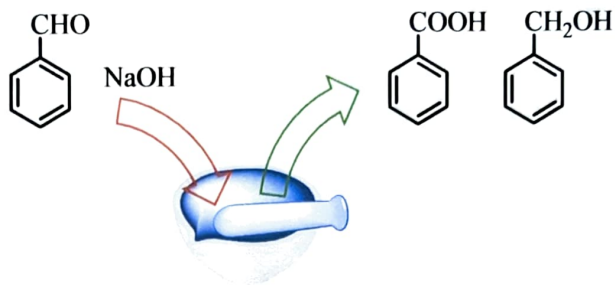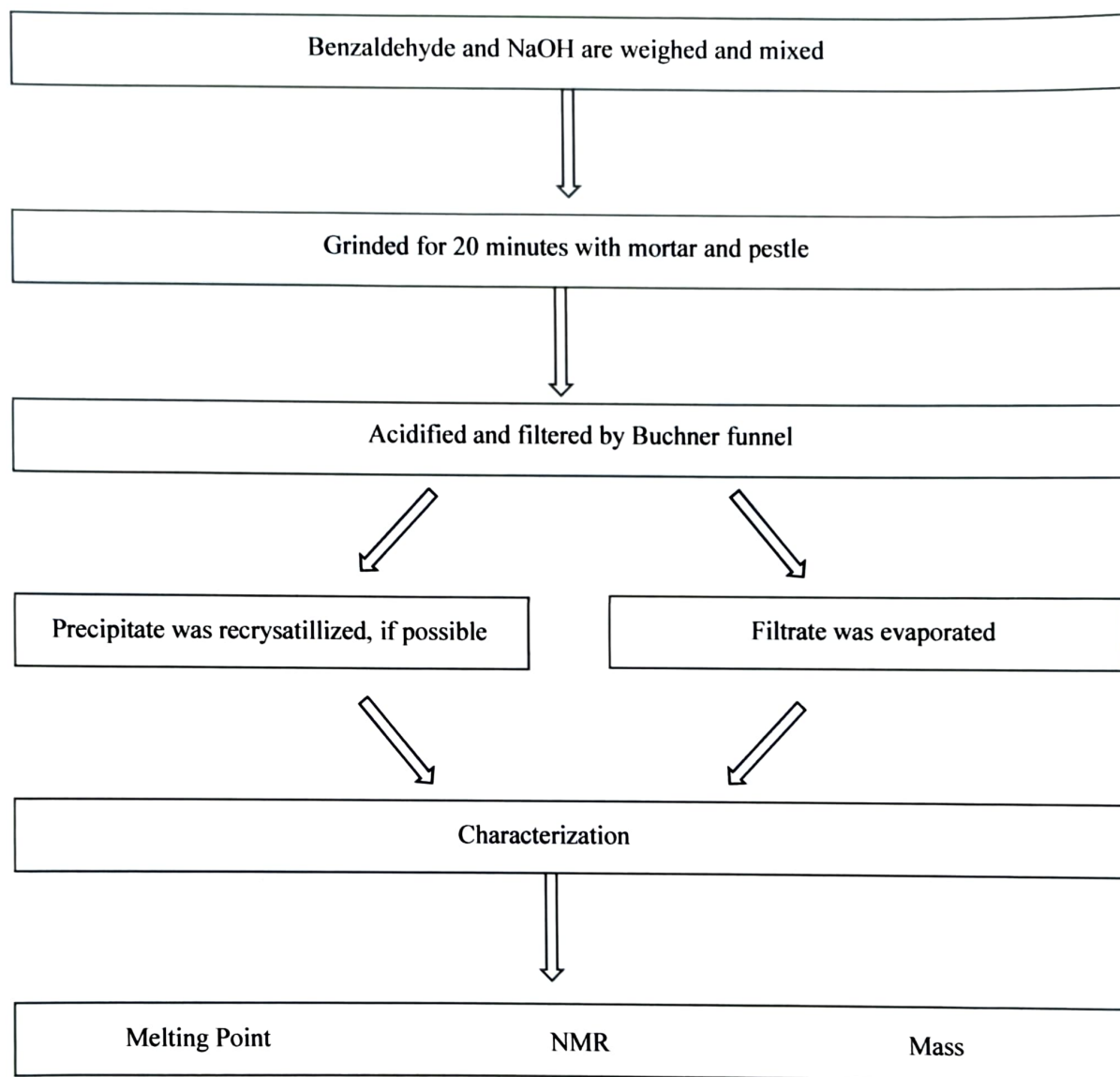
Chapter-1

1.1 INTRODUCTION

Cannizzaro reaction is one of the oldest known reactions in organic chemistry which involves the redox disproportionation of aldehydes with α-hydrogens in presence of strong base to give alcohol and carboxylic acids.[1] Despite its early occurrence, Cannizzaro reaction remains interesting to the synthetic chemists due to the synthetic and mechanistic challenges involved.[2] This reaction generally demands the use of strong basic conditions under heating. Over the years, different alkali and alkaline earth metal-based inorganic bases and organic bases also were explored in developing this reaction.[3] Additionally, this reaction was explored in presence of Lewis acid catalysts.[4] Nevertheless, Cannizzaro reaction has some inherent problems associated with the use of strong basic conditions under heating, which decrease the efficiency and hence, there is still scope of improvement in methodology development.

In recent times, people are more interested in developing reaction methodologies from the viewpoint of sustainability and application of solvent-free conditions is one of the major aspects in this direction. The advantages of such processes include the elimination of collecting-recycling step for solvents, attaining products often with high purity, thus eliminating the need of purification processes, lower energy requirement and lower processing cost. One of the main requirements of solvent-free reactions is to attain the desired molecular movement among the substrates. Unlike the reactions in solutions, where molecular movements are free, in solvent-free conditions, only the surface molecules of the solid components can come in contact with each other. One needs to disrupt the lattice structure of the solids in order to enable the maximum interaction between the molecules. In this direction, mechanochemical processes have evolved as a viable alternative in the 21st century. These processes mechanically break the molecular bonds by creating defects and dislocations in the lattice, reducing the particle sizes and increasing surface areas and generating local heating and melting. Mechanochemical processes include both grinding by mortar and pestle as well as use of ball mills. Although ball mills have the advantage of requiring no physical labour, grinding in mortar-pestle is operationally much simpler.[5] The use of mortar-pestle for organic transformations was introduced by Toda et. al. and often proceeds with solvent-free conditions without the need of a catalyst.[6] Thereby, this process was introduced gradually in different organic transformations such as from simple aldol condensation to peptide synthesis.

Cannizzaro reaction involves a hydride transfer mechanism which was found to be possible without the participation of the reaction medium.[3(a)] So, people anticipated that this reaction could be performed in a solvent-free medium under mechanochemical process. This report describes the Cannizzaro reaction of benzaldehyde derivatives under basic conditions using grinding in a mortar-pestle setup.[7]

FLOW CHART

| Benzaldehyde and NaOH are weighed and mixed |

| Grinded for 20 minutes with mortar and pestle |

| Acidified and filtered by Buchner funnel |

| Precipitate was recrysatillized, if possible | | Filtrate was evaporated |

| Characterization |

| Melting Point | NMR | Mass |

1.2 Present Work:

Cannizzaro reaction has been performed under solvent-free conditions using mortar & pestle with benzyldehyde, p-chlorobenzaldehyde and p- methylbenzaldehyde.

$$R = H, CH_3, Cl$$

<div align="center">**Chapter 2**</div>

2.1 Aim and Objective:

1. Green Synthesis of benzoic acid and benzyl alcohol derivatives by using benzaldehyde, *p*-chlorobenzaldehyde and *p*-methylbenzaldehyde as substrates.
2. Determination of melting point of the prepared compound.
3. Spectroscopic analysis of the product through 'H NMR and mass spectrometric techniques.

Chapter 3

3. Experimental details:
## 3.1 Requirements-

3.1.1 Apparatus:

1. Beakers
2. Filter paper
3. Glass rod
4. Measuring cylinder
5. Watch glass
6. Capillary tube
7. TLC plate
8. Buchner funnel
9. Test tube
10. Mortar and Pestle
11. Pyrex glass
12. Separating Funnel

3.1.2 Chemicals:

1. Benzaldehyde
2. P-chlorobenzaldehyde
3. P-methylbenzaldehyde
4. NaOH
5. Acetone
6. Chloroform
7. Hexane
8. HCl

3.2 Methodology :

Typical procedure: Benzaldehyde **A** (1 g, 9.4 mmol, 1 equiv.) and NaOH **B** (0.46 g, 11.32 mmol, 1.2 equiv.) were taken in a clean and dry mortar. This mixture was grinded well using pestle for 20 minutes at laboratoty temperature. The reaction proceeded exothermically. After completion of the reaction, as confirmed by TLC, 15 ml of ice –cold water was added to the mixture. The mixture was stirred. Then 2 M aqueous HCl solution was added to the mixture when white precipitate came. The precipitate was collected through filtration by Buchner funnel to get benzoic acid **C**. The filtrate was then extracted with chloroform and dried over anhydrous MgSO$_4$. The solvent was allowed to evaporate when benzyl alcohol **D** was obtained. The products were characterized by using melting point, whenever applicable, and spectroscopic techniques.
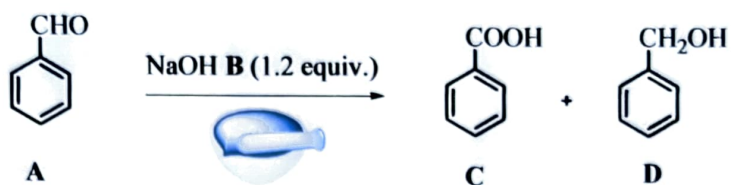
3.3 Diagrams of Consecutive steps in the experiment:
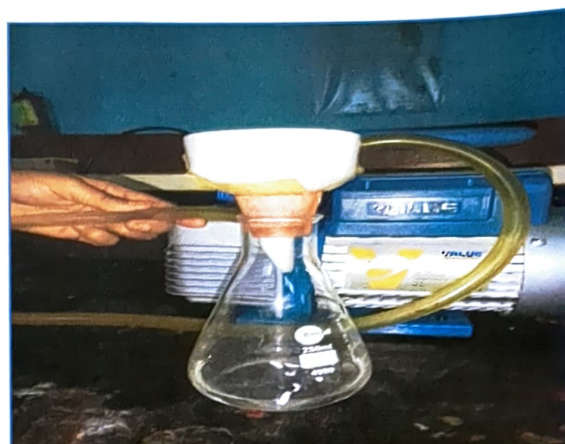

Fig: 1- Mixture of organic compounds
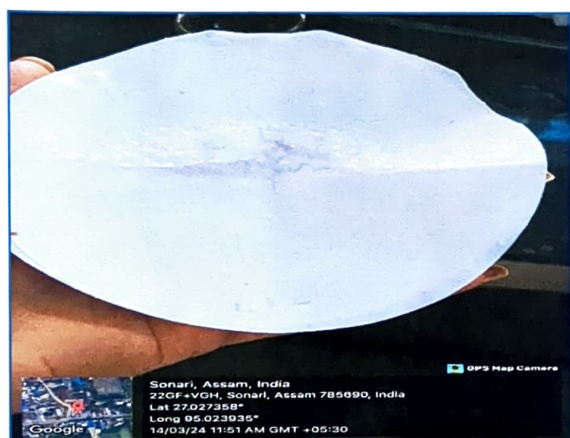

Fig: 2-Filteration by buchner funnel

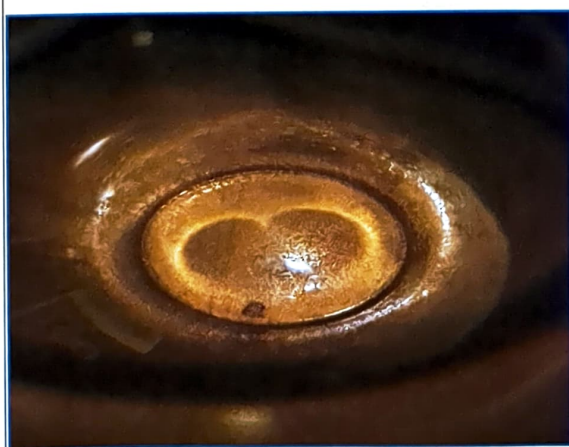Fig: 3- Formation of bezoic acid

Fig: 4- Separetion benzylalcohol

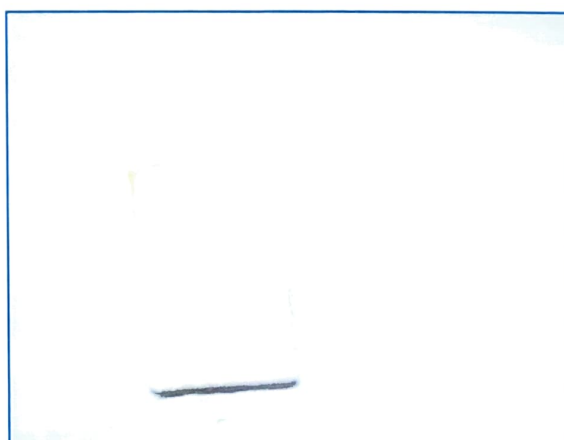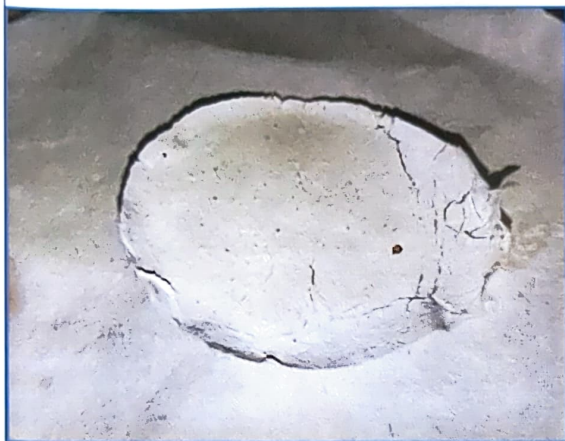Fig: 5- Formation of bezylalcohol

Fig: 6-TLC

Fig: 7- Formation of P-chloro benzoic acid
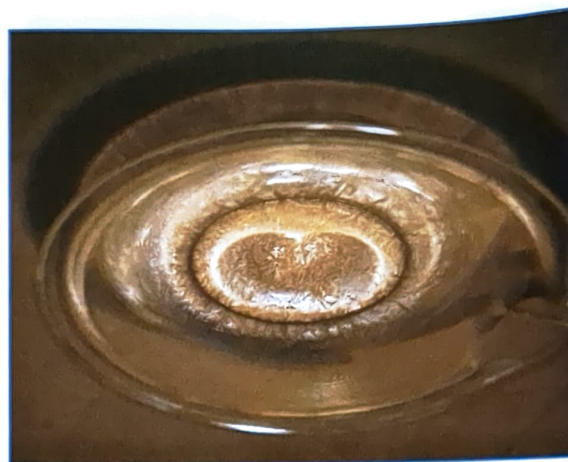

Fig: 8- Formation of P-chloro benzyl alcohol
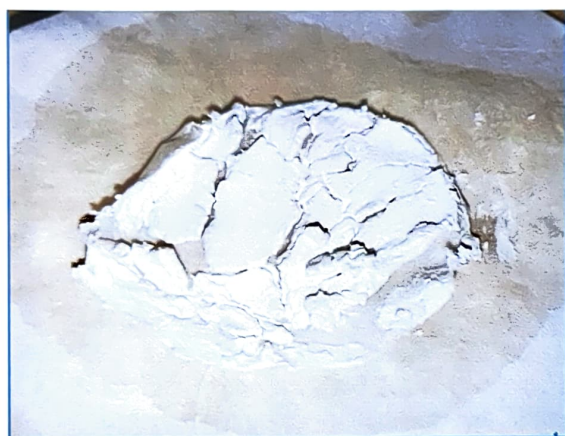
Fig: 9- Formation of P-methyl benzoic acid

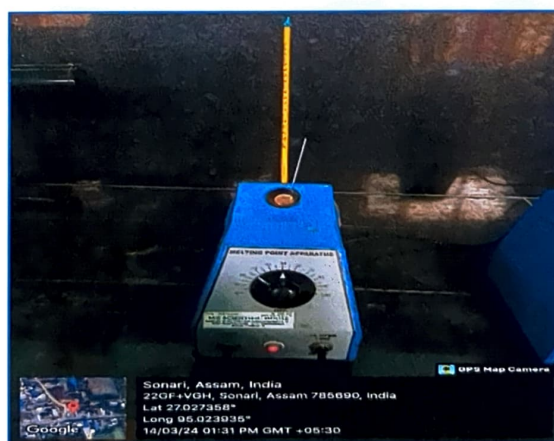Fig: 10- Formation of P-methyl benzyl alcohol

Sonari, Assam, India
22GF+VGH, Sonari, Assam 785690, India
Lat 27.027358°
Long 95.023935°
14/03/24 01:31 PM GMT +05:30

Fig: 12 Melting point
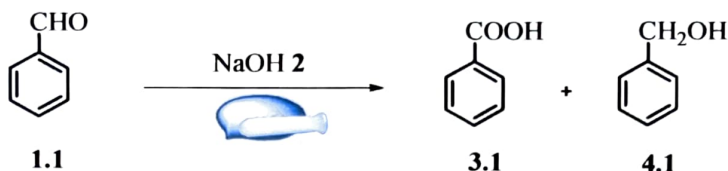
Result and discussion:

Reaction Optimization:

Table 1: optimization of reaction conditions

Sl. No	1.1 (equiv.)	2 (equiv.)	Percentage yield 3.1 (%)	Percentage yield 4.1 (%)
1	1	1	35	34
2	1	1.2	45	42
3	1	1.5	42	38

First, we set out to optimize the reaction conditions for maximum yield of the products. Benzaldehyde **1.1** was chosen as the model substrate for the transformation. We have only considered the yield of benzoic acid **3.1** for the optimisation process. First, we have used equimolar quantities of both aldehyde and NaOH and got 35% yield for benzoic acid **3.1**. Upon increasing the amount of base, we observed that maximum yield, i.e., 45%, was obtained when we used 1.2 equivalents of the base. On increasing the amount of the base further, the product yield decreased. This may be due to the excess amount of HCl used in the workup process which could interfere in the extraction process. We did not check other bases due to time constraints.

Table 2: substrate scope determination
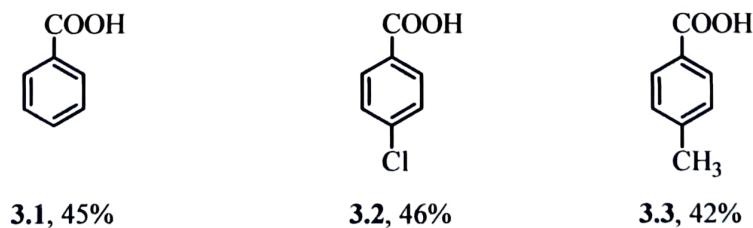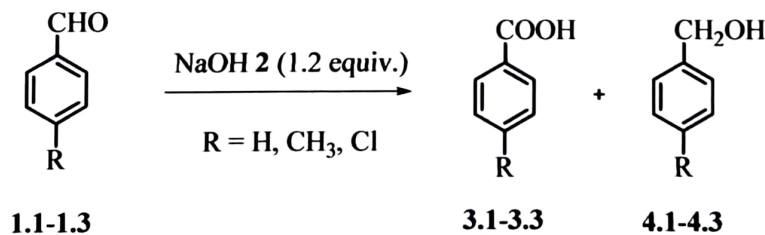
Experimental conditions: **1.1** (1 equiv.), **2** (1.2 equiv.), mortar-pastle, 20 min, RT

Upon establishing the optimized reaction conditions, we extended the substrate scope to two other benzaldehyde derivatives, *viz.* *p*-chlorobenzaldehyde **1.2** and *p*-methylbenzaldehyde **1.3**. The transformation was found to be 100% in all the cases. The yield of **3.3** was found to be slightly less (42%) than **3.1**, whereas, that of **3.2** was found to be almost equal. Since the yield difference was not much, we did not think that it might be due to the electronic effect, although, electron-withdrawing effect can increase the rate of Cannizzaro reaction.

4.2 Characterization:

4.2.1 - Thin layer Chromatography

TLC plate was prepared by using a uniform coating of silica gel G over the glass plate which was dried in hot air oven. The sample was dissolved in acetone and the spot was applied on the TLC plate with the help of capillary tube. Then the TLC plate was run by using methanol and heaxane (1:10) as eluent in a closed container. When the solvent front has nearly reached the top of the stationary phrase, the plate was removed from the container and the solvent front was marked with a pencil. The spot was visualized by inserting the TLC plate in an iodine chamber. After removing the plate, the distance travelled by the compound was marked. From this, the retention factor or R_f value can be calculated.[ref]

4.1:-

$$R_f = \frac{\text{Distance travelled by the solute}}{\text{Distance travelled by the solvent}}$$

$$= \frac{4.5}{5}$$

$$= 9$$

Solvent font

5cm

4.5cm

Spot of Sample
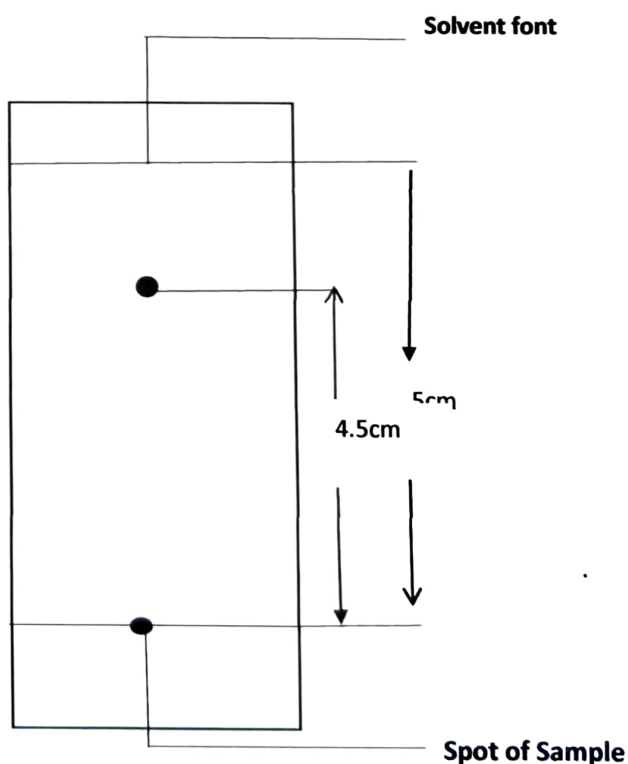
4.1: The retention factor or R_f value of benzylalcohol is 0.9

4.2: The retention factor or R_f value of p-chlorobenzyl alcohol is 0.4

4.3: The retention factor or R_f value of p-methylbenzeldehyde is 0.45

4.2.2 Appearance: - The benzoic acid derivatives **3.1 – 3.3** were found to be crystalline solids. On the other hand, the benzylalcohol derivatives **4.1 – 4.3** were found to be white powde.

4.2.3 Determination of melting point:- A small amount of the prepared sample is introduced in a capillary tube and its melting point is determined.

3.1: The compound was found to be melted at 120 °C which resembles the melting point of Benzoic acid (121-122 °C)[ref]. This indicates that the product **3.1** should be benzoic acid.

3.2: The compound was found to be melted at cannot be determined which resembles the melting point of Benzoic acid (241°C)[ref]. This indicates that the product 3.2 should be benzoic acid.

3.3: The compound was found to be melted at 175 °C which resembles the melting point of Benzoic acid (177-180°C)[ref]. This indicates that the product **3.1** should be benzoic acid.

4.2.4: Yield calculation:

CHO

NaOH **2** (1.2 equiv.)

R = H, CH₃, Cl

COOH   CH₂OH

+

R   R

1.1 - 1.3   **3.1 - 3.3**   **4.1 - 4.3**
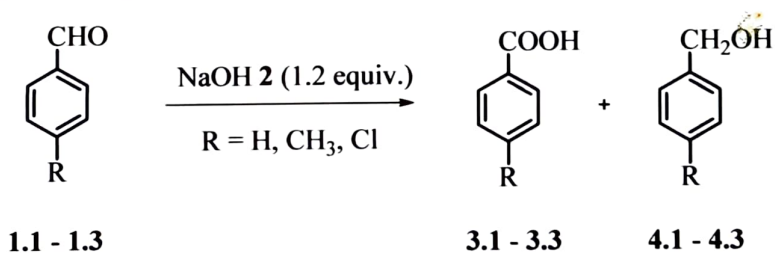
When   **1.1** = Benzaldehyde

2 = NaOH

3.1 = Benzoic acid

4.1 = benzylalcohol

	1.1	**2**
Molecular weight	106 .12 g/mol	40 g/mol
Weight taken	1g	0.46 g
mmol	9.4	11.32
Equivalent	1	1.2

Product yield for **3.1** = 0.52 g = 520 mg

mmol of **3.1** = 510/122.12 = 4.2

% of yield for **3.1** = mmol of **3.1** obtained / mmol of reactant **1.1**

$$= 4.2/9.4 \times 100\%$$

$$= 0.45 \times 100\%$$

$$= 45\%$$

Product yield for **4.1** = 0.39=390mg

mmol of **4.1** = 390/108=3.6

% of yield for **4.1** = mmol of **4.1** obtained / mmol of reactant **1.1**

$$= 40/9.4 \times 100\%$$

$$= 0.42 \times 100\%$$

$$= 42\%$$

1. Where, 1.2= Benzaldehyde

    3= NaOH

    3.2= p-cholorobenzoic acid

    4.2=p-chlorobenzylalcohol

	1.2	3
Molecular weight	140.57g/mol	40g/mol
Weight taken	1g	0.34g
mmol	7.14	8.6
Equivalent	1	1.2

Product yield for **3.2** = 0.47g = 470 mg

mmol of **3.2** = 3.3/ 140= 3.5

% of yield for **3.2** = mmol of **3.2** obtained / mmol of reactant **1.2**

$$= 3.3/7.14 \times 100\%$$

$$= 0.46 \times 100\%$$

$$= 46\%$$

Product yield for **4.2** = 0.42=420mg

mmol of **4.2** = 420/142=2.9

% of yield for **4.2** = mmol of **4.2** obtained / mmol of reactant **1.2**

$$= 2.9/7.14 \times 100\%$$

$$= 0.40 \times 100\%$$

$$= 40\%$$

1. Where, 1.3=p-methylbenzaldehyde

    4= NaOH

    3.3= p- methylbenzoic acid

    4.3=p-methylbenzylalcohol

	1.3	4
Molecular weight	120g/mol	40g/mol
Weight taken	1g	.40
mmol	8.3	10
Equivalent	1	1.2

Product yield for **3.3**= 0.42g = 420 mg

mmol of **3.3**= 420/120 = 3.5

% of yield for **3.3** = mmol of **3.3** obtained / mmol of reactant **1.3**

$$= 3.5/8.3 \times 100\%$$

$$= 0.42 \times 100\%$$

$$= 42\%$$

Product yield for **4.3** = 0.39=390mg

mmol of **4.3** = 390/122=3.1

% of yield for **4.3** = mmol of **4.3**obtained / mmol of reactant **1.3**

$$= 3.1/8.3 \times 100\%$$
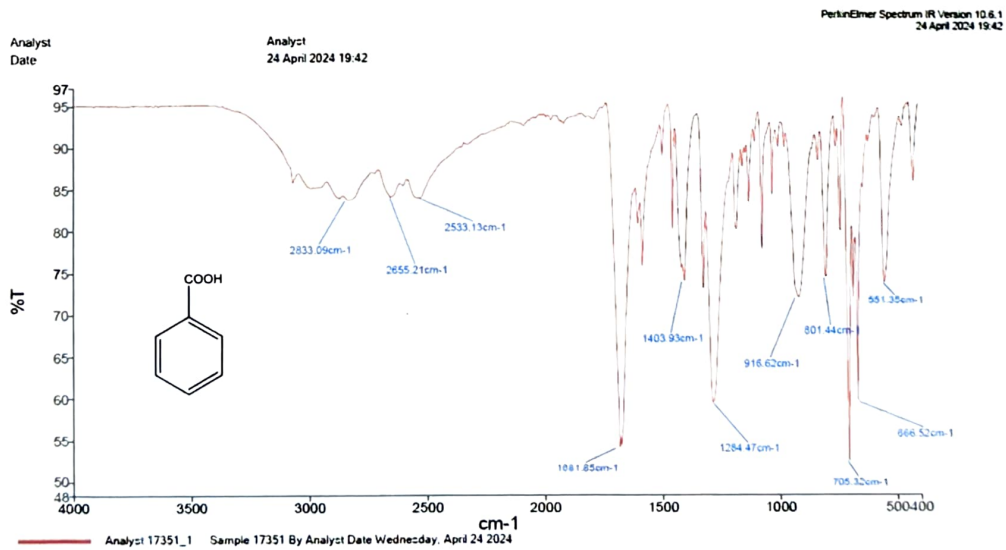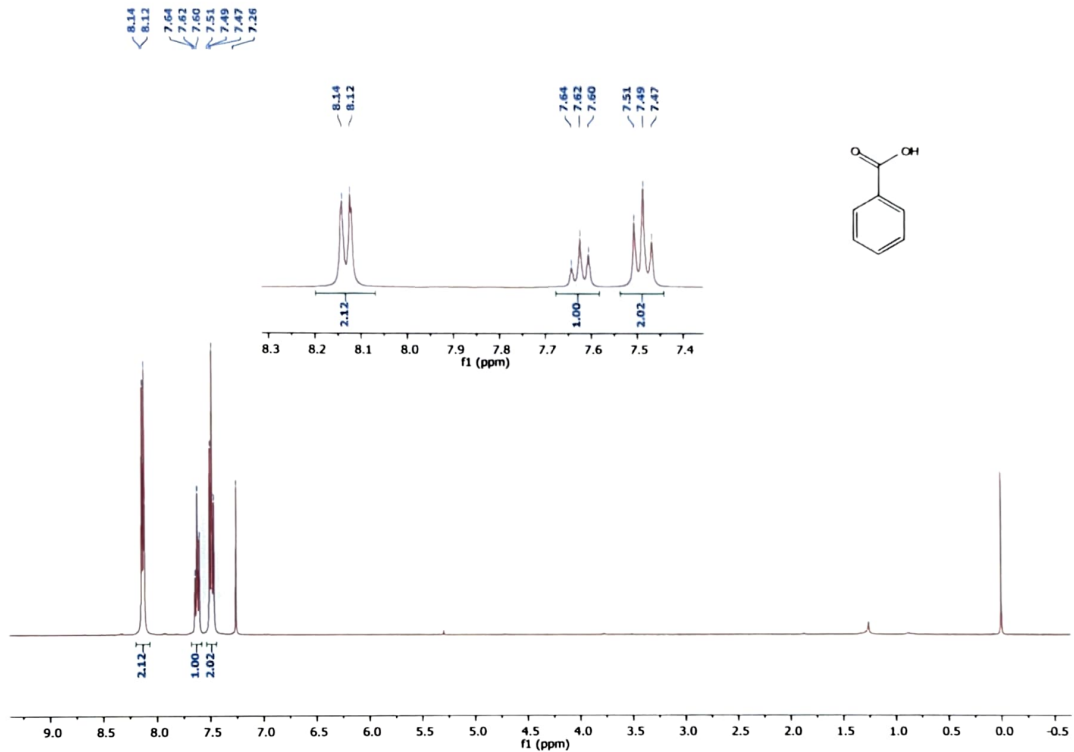
$$= 37 \times 100\%$$

$$= 37\%$$

Table 3: Physical characteristics of Product:-

SL NO.	Products name	Product color	Product smell	Physical state	solubility	Melting point
1.	Benzoic acid	White crystal	Odorless	Crystalline solid	Soluble in water	$120^{o}C$
2.	p-chclorobenzoic acid	White crystal	Odorless	Crystalline solid	Soluble in ethanol	Cannot be determined
3.	p-methylbenzoic acid	White crystal	Odorless	Crystalline solid	Soluble in acetone	$175^{o}C$

SL NO.	Products name	Product color	Product smell	Physical state	solubility	Melting point
crystalline	Benzaylalcohol	colourless	Faint aromatic odor	White powder	Soluble in water	$-15^{o}C$
crystalline	p-chlorobenzyalcohol	White	Faint aromatic odor	White powder	Soluble in methanol	58-61
crystalline	p-methylbenzylalcohol	White	Mild floral odor	White powder	Soluble in ether and ethanol	57-61

4.2.5 Observation of NMR:-

The 'h NMR spectrum of the prepared Benzoic acicdis recorded using NaOH, a solvent at room temperature. It is represented below:

^{1}H NMR spectrum

Peaks: 8.05, 8.04; 7.31, 7.30; 2.46

f1 (ppm)

Integrations: 2.00, 2.00, 3.00
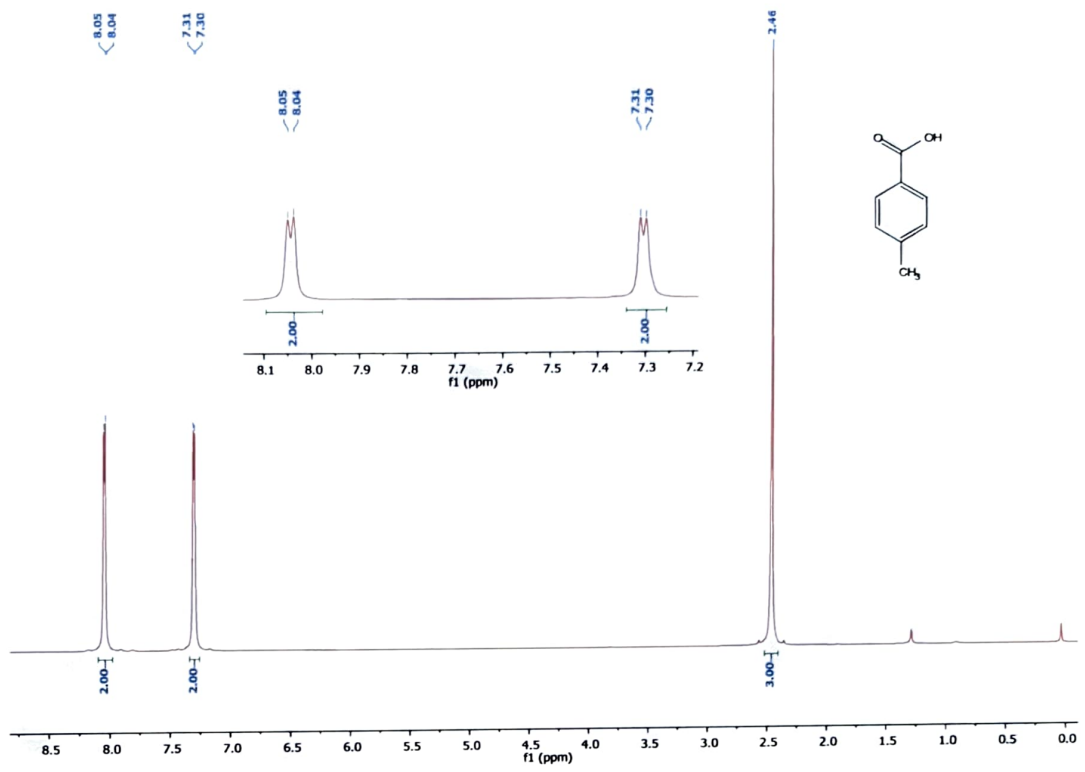
PerkinElmer Spectrum IR Version 10.6.1
24 April 2024 19:58

Analyst
Date

Analyst
24 April 2024 19:58

IR spectrum (%T vs cm-1)

2532.35cm-1
1668.52cm-1
1610.90cm-1
1416.81cm-1
1281.97cm-1
1182.52cm-1
1116.88cm-1
960.18cm-1
839.17cm-1
752.82cm-1
607.51cm-1
540.84cm-1
467.61cm-1
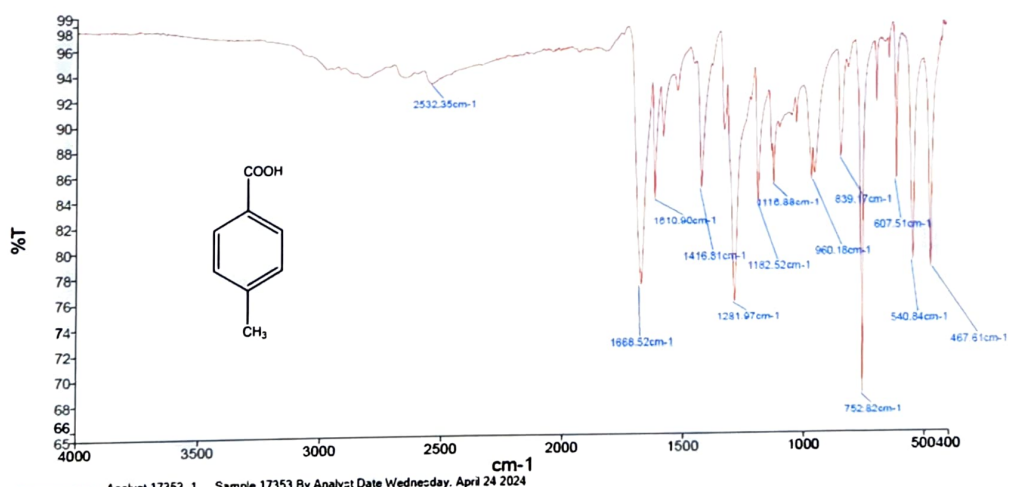
Analyst 17353_1   Sample 17353 By Analyst Date Wednesday, April 24 2024

NMR spectrum peaks: 7.25, 7.23, 7.17, 7.15, 4.61, 2.34

Inset: 7.25, 7.23, 7.17, 7.15

f1 (ppm): 7.35 7.30 7.25 7.20 7.15 7.10 7.05 7.00 6.95

Integrations: 1.89, 1.67, 1.83, 3.00

f1 (ppm): 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0

PerkinElmer Spectrum IR Version 10.6.1
24 April 2024 19:51

Analyst
Date

Analyst
24 April 2024 19:51
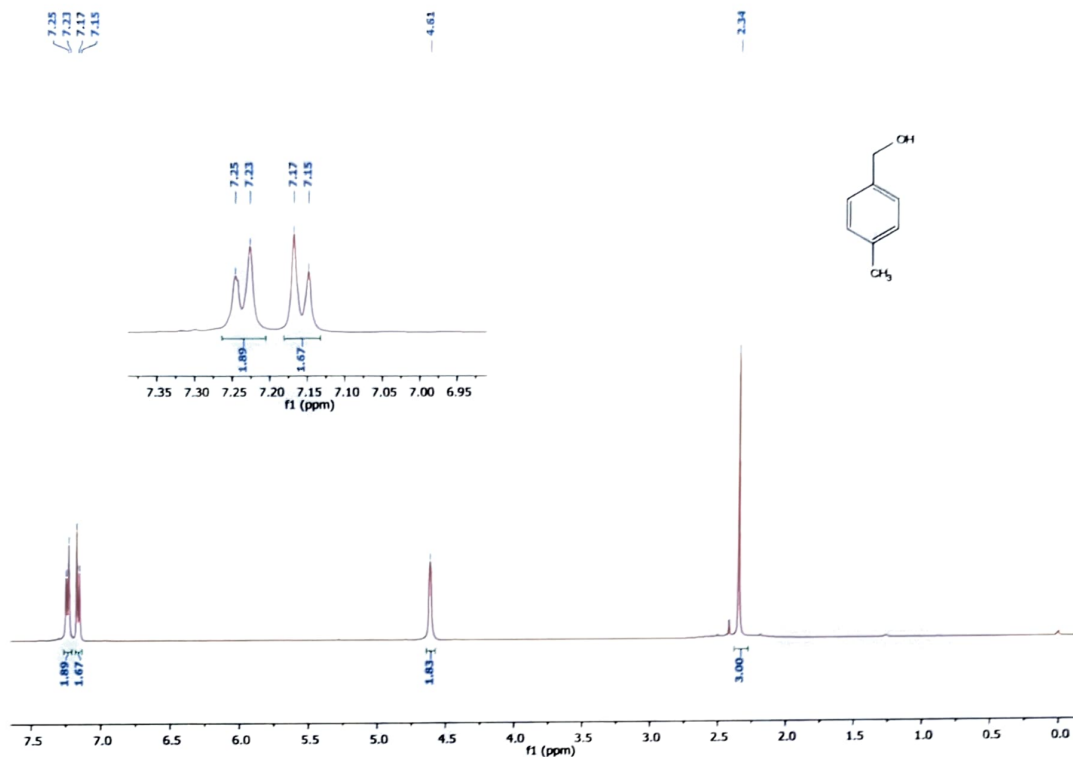

IR spectrum (%T vs cm-1):
3347.28cm-1
2920.23cm-1
2852.70cm-1
1901.77cm-1
1668.78cm-1
1611.48cm-1
1517.07cm-1
1414.88cm-1
1282.31cm-1
1178.24cm-1
1010.49cm-1
802.47cm-1
753.31cm-1
607.79cm-1
468.20cm-1

%T axis: 97 96 94 92 90 88 86 84 82 80 78 76 74 72 70 68 66 64 62

cm-1 axis: 4000 3500 3000 2500 2000 1500 1000 500 400

Analyst 17352_1   Sample 17352 By Analyst Date Wednesday, April 24 2024

IR spectrum with labeled peaks:
- 2561.42cm-1
- 1679.09cm-1
- 1590.94cm-1
- 1423.36cm-1
- 1281.99cm-1
- 1175.65cm-1
- 1091.21cm-1
- 1015.34cm-1
- 924.83cm-1
- 852.01cm-1
- 760.23cm-1
- 682.16cm-1
- 546.34cm-1
- 471.85cm-1

Structure: 4-chlorobenzoic acid (COOH on benzene ring with Cl)
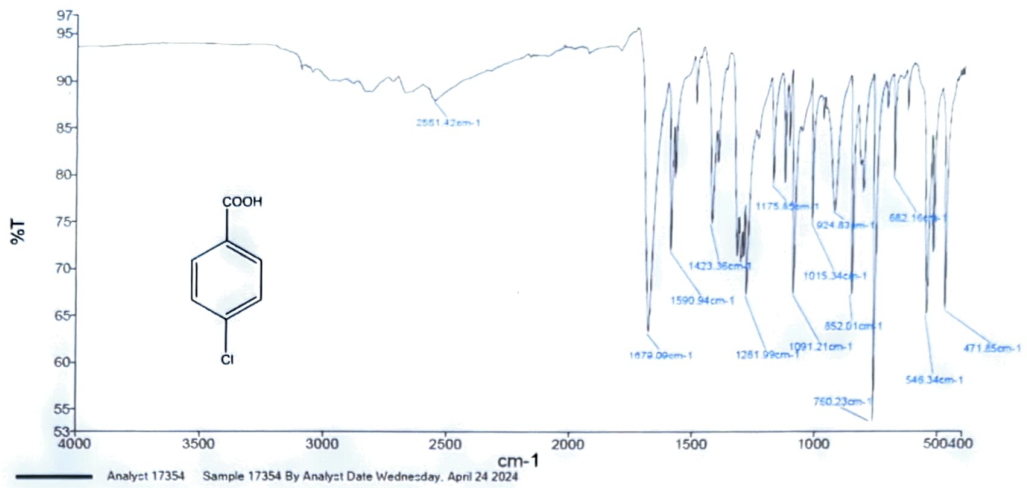
Analyst 17354   Sample 17354 By Analyst Date Wednesday, April 24 2024

8.06
8.05
7.48
7.47
7.28

0.75
2.00
1.94

11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

NMR peaks: 7.33, 7.31, 7.29, 7.27, 7.26 — 4.65 — 2.06 — 0.00

Expansion: 7.33, 7.31, 7.29, 7.27, 7.26

f1 (ppm)

Integration: 4.10, 2.00

PerkinElmer Spectrum IR Version 10.6.1
24 April 2024 20:08
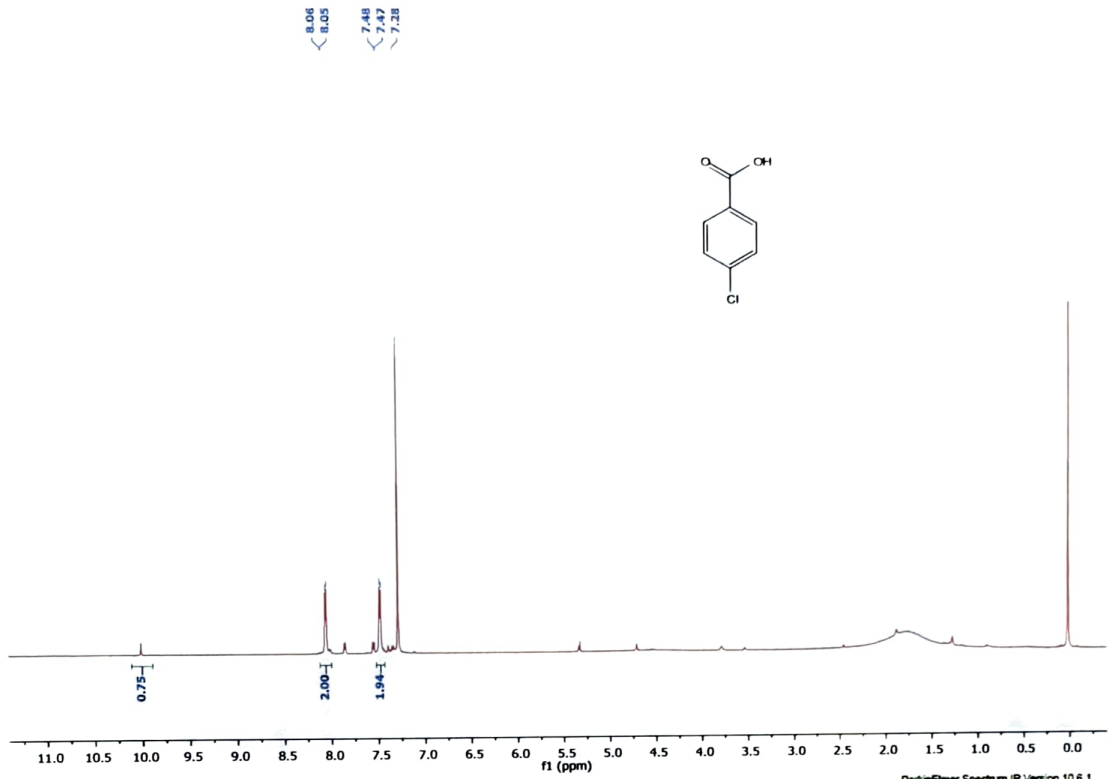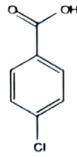
Analyst
Date

Analyst
24 April 2024 20:08

%T vs cm-1

IR peaks: 3335.10cm-1, 2922.06cm-1, 1678.76cm-1, 1596.58cm-1, 1490.71cm-1, 1403.42cm-1, 1207.22cm-1, 1084.98cm-1, 1005.24cm-1, 829.73cm-1, 797.62cm-1, 652.66cm-1, 519.10cm-1, 445.28cm-1

Analyst 17355_1   Sample 17355 By Analyst Date Wednesday, April 24 2024

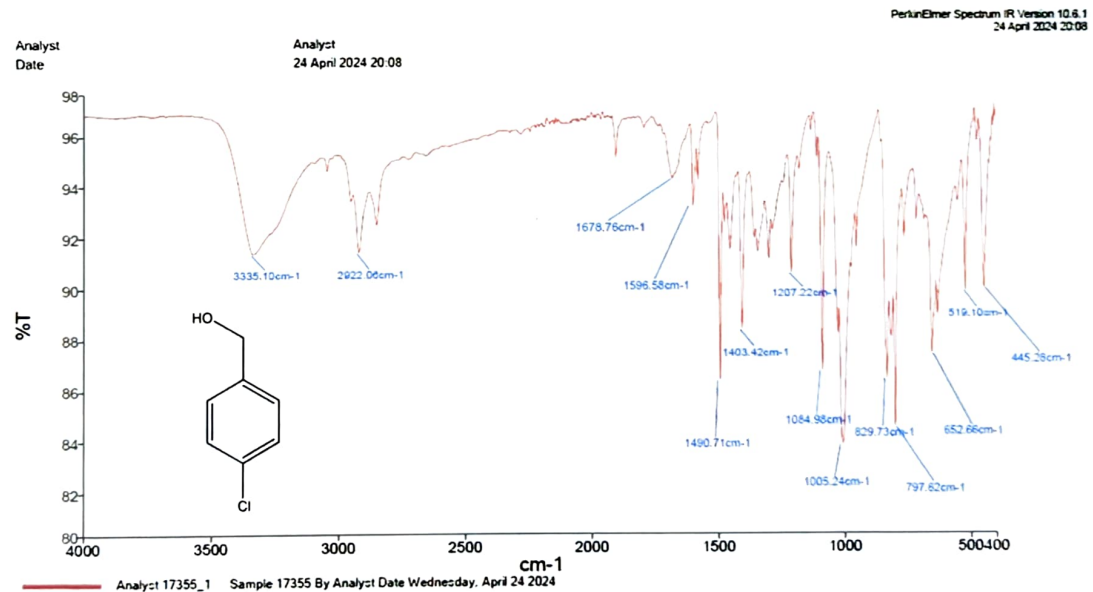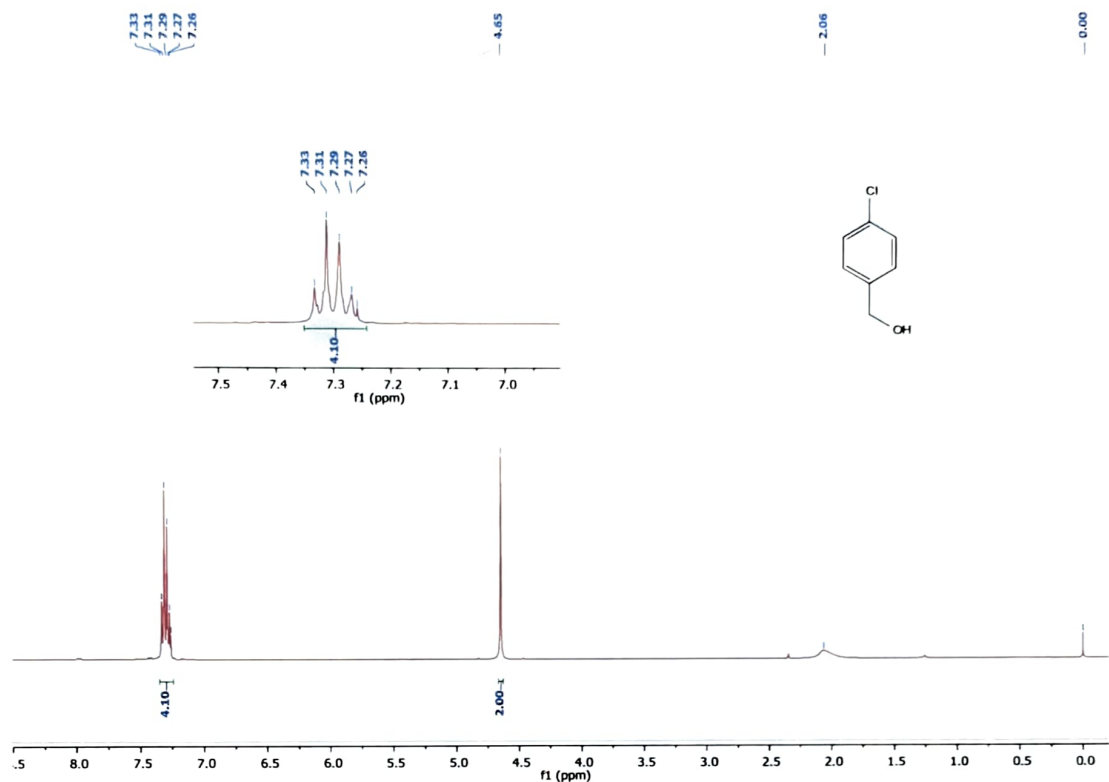## 4.2.6 Mechanism:-

Scheme: mechanism of Canniczaro Reaction

Chapter 5:

Conclusion:

In conclusion, a simple and efficient procedure for the Cannizzaro reaction was established. This present method is superior since it is eco-friendly, advantageous over previously described methods in yield, requires no special apparatus, there is simplicity of operation, and is non-hazardous, simple and convenient.

Chapter 6:

Reference:

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8. .Selective reduction of aldehydes and ketones to alcohols with ammonia borane in water
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10. Nickel-catalyzed carboxylation of aryl iodides with lithium formate through catalytic CO recycling

Mr Amrit Kumar Borpujari   Date:

Associate Professor, Dept. of Chemistry(HOD)

CERTIFICATE

This is to certify that the project work entitled "*Analiysis of Phisico-Chemical Parameter of soil of differrent localities of Charaideo district*" submitted by Anindya Akash Phukon, Nitish Kumar Borgohain and Monuj Kharia students of 6th sem of the Department of Chemistry, Sonari affiliated to Dibrugarh university is carried out under my supervision and is found suitable to submitted the partial fulfillment of the B.Sc. Chemistry. This work in the present form in part, has not been submitted for any purpose elsewhere.

I wish his great success in future.

(Amrit Kumar Borpujari)

Place: Sonari College

...t of Chemistry

...e, Sonari

...785690, Assam, India

Dr Bikash Kumar Sarmah

Assistant Professor, Dept. of ChemistryDate:

CERTIFICATE

This is to certify that the project work entitled **Solvent Free "Cannizzaro reaction by grind stone technique"** Submitted by Lovita Teli a student of 6th semester to the Department of Chemistry, Sonari College, Sonari, affiliated to Dibrugarh University is carried out under my supervision and is found suitable to submit for the partial fulfillment of the B.Sc. Degree in Chemistry. This work, in the present form or in part, has not been submitted for any purpose elsewhere.

I wish her great success in future.

Signature of Guide

(Dr. Bikash Sarmah)

Place: Sonari College

Suchitra Narayan Rajkhowa

Date: 22/05/24

Associate Professor(Dept. of Chemistry)

CERTIFICATE

This is to certify that the project work entitled "To analysis the physico- chemical parameter of three ancient ponds of Sonari" submitted by Mayuri Gogoi, a student of 6th sem of the Department of Chemistry, Sonari affiliated to Dibrugarh university is carried out under my supervision and is found suitable to submitted the partial fulfillment of the B.Sc. Chemistry. This work in the present form in part, has not been submitted for any purpose elsewhere.

I wish her great success in future.

S. N. Rajkhowa

Signature of Guide

Suchitra NarayanRajkhowa

Place: Sonari College