

**MANNICH AMINOMETHYLATION REACTION DIETHYL SUCCINATE AS A  
CARBON ACID**

Submitted in partial fulfillment of the requirements for the award

Of

Master of Science Degree in Chemistry

By

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Register No: 39910016



DEPARTMENT OF CHEMISTRY

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SATHYABAMA INSTITUTE OF SCIENCE AND TECHNOLOGY

(DEEMED TO BE UNIVERSITY)

Accredited with Grade "A" by NAAC | 12B Status by UGC | Approved by AICTE

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**DEPARTMENT OF CHEMISTRY**

**BONAFIDE CERTIFICATE**

This is to certify that this Project Report is the bonafide work of **M.PREMKUMAR (39910016)** who carried out the project entitled "**MANNICH AMINOMETHYLATION REACTION DIETHYL SUCCINATE AS A CARBON ACID** " under my supervision from December 2020 to March 2021.

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Submitted for Viva voce Examination held on\_\_\_\_\_

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External Examiner

## DECLARATION

I, **M.PREMKUMAR**, hereby declare that the Project Report entitled “**MANNICH AMINOMETHYLATION REACTION DIETHYL SUCCINATE AS A CARBON ACID**” done by me under the guidance of **Dr.S. SUPRIYA M.Sc.**, M. Phil, Ph.D., Associate Professor, Department of Chemistry, Sathyabama Institute of Science and Technology is submitted in partial fulfilment of the requirements for the award of Master of Science degree in Chemistry.

DATE:

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M.PREMKUMAR

## **ABSTRACT**

Mannich aminomethylation products have been prepared by one pot synthesis the complex with formaldehyde and diethyl succinate. the complex was characterized by suitable spectroscopic studies and analytical methods such as uv-visible spectroscopy infrared spectroscopy , elemental analysis and mass spectroscopy .mannich base synthesis medicinal value and anti-inflammatory, antibacterial , anticonvulsant , antiviral activities in adding other bioactive applications. Mannich base synthesis using metal complex , we have successfully synthesized a mannich base reaction metal amino acid and using diethyl succinate as a carbon acid .

Key words: diethyl succinate , mannich aminomethylation , mannich base synthesis , Density calculation.

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# CHAPTER -1

## INTRODUCTION

### 1.1 COPPER METAL COMPLEXES

Their structures as been elucidated on the basis analytical ,magnetic, electrical conductivity and spectral study as well as elemental analyses . the complexes exhibit square-planar geometry. copper exhibits considerable biochemical action either as an essential trace metal or as a constituent of various exogenously administered compound in humans. I while in its latter it is bound to various types forming complexes that interact with biomolecules, mainly proteins and nucleic acid . He biochemical action of Cu complexes with non-steroidal anti- inflammatory drugs. Enhanced anti- inflammatory and antiulcerogenic activity,as well as reduced gastrointestinal toxicity compared to the uncomplexed drug , have been prepared and structurally characterized .

Copper exhibits considerable biochemical action either as an essential trace metal or as a constituent of various exogenously administered compound in humans .have brought to attention the antiviral and antibacterial activity of cu(II) complexes. For instance , it was shown that the infectivity of influenza A virus is reduced after exposure on copper surfaces . The study and development of Cu complexes could be helpful in the desing and production antiviral and antibacterial materials . despite the face that the action of copper in humans has been intensively studied , the clinical picture of copper status is not always so straightforward , and less is know about the role of copper complexes in medicine . copper in food is processed by the liver and transported and sequesteres in a safe manner . inorganic copper , such as thet in drinking water and copper supplements , largely bypasses the liver and enters the free copper pool of the blood directly . this copper is potentially toxic because it amy penetrate the blood/brain barrier.

### 1.2 AMINO ACID

Amino acid are molecules containing an amine group, a carboxylic acid group and aside chain that varies between different amino acid these molecules are particularly important . metal ion form a significant portion of the active site of biomolecule such as proteins and enzymes to accomplish evolutionary directed functions . complexes between a number of metals and aminoacids containing only two ionizing group . periodic classification were studied in their relation to glycine , its derivatives and other amino acid with only two ionizing group . in this paper an attempt has been

made to study the effect on complex formation of (a) the nature of the metal , and (b) substitution in the amino-acid. amino acid forms the building blocks of proteins and an essential role in the metabolism and regulation of living organism. Nine proteinogenic amino acid , called essential amino acid , are essential for humans because they cannot be produced . one of the major research areas of reactions involving amino acid is in condensation reaction in mannich aminomethylation with carbon acid such as diethyl succinate .thus, it is the aim of the study to explore the effect of pH and reaction condition for mannich aminomethylation reaction with chelated glycinate Cu(II) complex.

### **1.3 COPPER AMINO ACID COMPLEX;**

Copper researches in these areas focus on the synthesis and characterization of biological compounds containing metal ions due to their applicability in pharmacy , medicine , agronomy and nutrition . The modern chemotherapy is promoted on the basis of metal and metal complexes. The complexes formed by the simplest amino acid , glycine , with different bare and hydrated metal ions  $\text{Cu}^{2+}$  were studied in the gas phase and in solvent in order to give better insight into the field of the metal ion-biological ligand interactions. The effect of the size and charge of each cation on the organization of the surrounding water molecules were analyzed.

Glycine is an amino acid that your body uses to create proteins , which it needs for the growth and maintenance of tissue and for making important substance , such as hormones and enzyme . glycine is an amino acid that has hydrogen atoms as its side chain . glycine is one of the proteinogenic amino acid , glycine used to memory and thinking skills(cognitive function ) .early research shows that taking glycine by mouth might improve and mental performance .

#### 1.4 MANNICH AMINOMETHYLATION REACTION

Mannich aminomethylation reaction become synthesis of macrocyclic compound and complexes , many of the mannich base compounds synthesized by the condensation of a secondary amine and cyclic imide have been recorded. The ring closing agents used as active hydrogen containing compound by nitroalkane, In the particular instance when formaldehyde is employed as aldehyde component , the substrate is converted into the corresponding mannich base through an aminomethylation process. Formaldehyde and diethyl succinate result in the respective metal(II) complexes of glycine , which are characterized by various physical means , including X-ray structural analyses of the zinc(II) and nickel(II) complexes

The products formed were found to vary with the PH and the alky substituent of the chelated amino acid used in mannich aminomethylation products and their application . mannich aminomethylation reaction of chelated amino acid complexes using diethyl succinate as the carbon acid in the literature . Hence the work mannich aminomethylation reaction of bis(glycinato) copper(II) complexes with formaldehyde and diethyl succinate as the carbon acid .

## CHAPTER -2

### LITERATURE SURVEY

Gheorghe Roman

Mannich base reaction it provides an updated and comprehensive look at these compound –compounds identification. The biological activity of mannich bases a structurally heterogeneous class of compound that are generated from various substrates through the introduction of an aminomethyl function by means of the mannich reaction .

The review is devoted to the aminomethylation of the CH-,NH-and the PH- acid residues by aminoacid . data are presented on the biological activities of certain mannich bases derived from aminoacid

Mannich aminomethylation reaction is surveyed , with emphasis on the relationship between structure and biological activity . The review covers extensively the literature report that have disclosed mannich bases as anticancer and cytotoxic agents , or compounds with potential antibacterial and antifungal activity in the last decade . The most relevant studies on the activity of mannich bases as antimycobacterial agents , antimalarials , anti-inflammatory , analgesic and antioxidant activities of mannich bases

As well as their use as agents for the treatment of mental disorders have been presented . the review gives in the end a brief overview of the potential of mannich bases as inhibitors of various enzymes or ligands for several receptors .

Angelo clerici, Rosalba canella,nadia pastori tert – Butoxy, generated by Ti(III) one electron reduction of tert-butylhydroperoxide, selectively abstracts an ether. One pot aminomethylation of ether at room temperature.

## **CHAPTER 3**

### **AIM AND SCOPE OF THE WORK**

The aim of the reaction to optimize condition for the mannich aminomethylation reaction of the  $\text{Cu(II)glycinato}$  complex using formaldehyde and diethyl succinate as a carbon acid. The interpretation of the formed mannich base complexes calculations metal ion determined. The scope of this work to evaluate the biological activity of the formed mannich base product and applications catalytic activity in different reaction processes.

### **SCOPE**

Mannich aminomethylation reaction using formaldehyde and diethyl succinate

Conformation of the product by the following characterization ( UV , FTIR )

Testing for antibacterial activity of the following product

## CHAPTER 4

### EXPERIMENTAL PROCEDURE

Glycine , aqueous solution of formaldehyde and diethyl succinate ,

#### 4.2 MATERIAL AND MEASUREMENT;

UV/VIS spectroscopy is routinely used in analytical chemistry for the quantitative determination of different analytes , such as transition metal ions , highly conjugated organic compound , and biological macromolecules . spectroscopic analysis is commonly carried out in solution but solids and gases may also be studied . solution of transition metal ions can be colored of the chemical involved. In this region of the electromagnetic spectrum atoms and molecules undergo electronic transition. Absorption spectroscopy is complementary to fluorescence deals with transitions from the excited state to the ground state, while absorption measure transitions from the ground state to the excited state.



Fig 4.2 UV spectrometer

### 4.3 FTIR ( FOURIER TRANSFORM INFRARED SPECTROSCOPY)

Fourier transform infrared spectroscopy is a technique used to obtain an infrared spectrum of absorption or emission of a solid, liquid or gas. The method or technique of infrared spectroscopy is conducted with an instrument called an infrared spectrophotometer to produce an infrared spectrum. The basic theory at work is the bonds between different element absorb light at different frequencies. The light is measured using an infrared spectrometer which produces the output of an infrared spectrum. The measure intensity over a narrow range of wavelength at a time. An IR spectrum can be visualized in a graph of infrared light absorbance on the vertical axis vs frequency or wavelength on the horizontal axis. spectroscopy is the study of interaction of the sample were qualitatively analyzed by FTIR spectroscopy and record the range from 400-4000  $\text{cm}^{-1}$ .

A laboratory instrument that uses this technique is a fourier transform infrared spectroscopy or spectrometer.



Fig 4.3 fourier transform infrared spectrometer

### **4.3.1 RANGE OF IR SPECTROSCOPY**

The infrared spectrum was recorded on a FT-IR spectrophotometer from the range of 400-4000  $\text{cm}^{-1}$  as KBr pellet and are wave numbers. The IR complex shows peaks at 439, 475, 509, 588, 694, 739, 856, 930, 988, 1033, 1154, 1226, 1314, 1362, 1453, 1478, 1608, 3190 this based on the IR peaks the following structure has been assigned for the synthesized product.

### **4.4.1 PREPARATION OF MANNICH AMINOMETHYLATION COMPLEX**

Dissolved 0.5g of glycinate complex 50ml of distilled water. The solution was taken in a clean round bottom flask. Take formaldehyde 1ml and diethyl succinate 1.28ml added dropwise using burette with constant stirring on magnetic stirrer. Maintain the temperature in  $60^{\circ}\text{C}$  the reaction continuously in 4 days. The product form in pale blue colored the product filtered by whatmann filter paper.

## CHAPTER -5

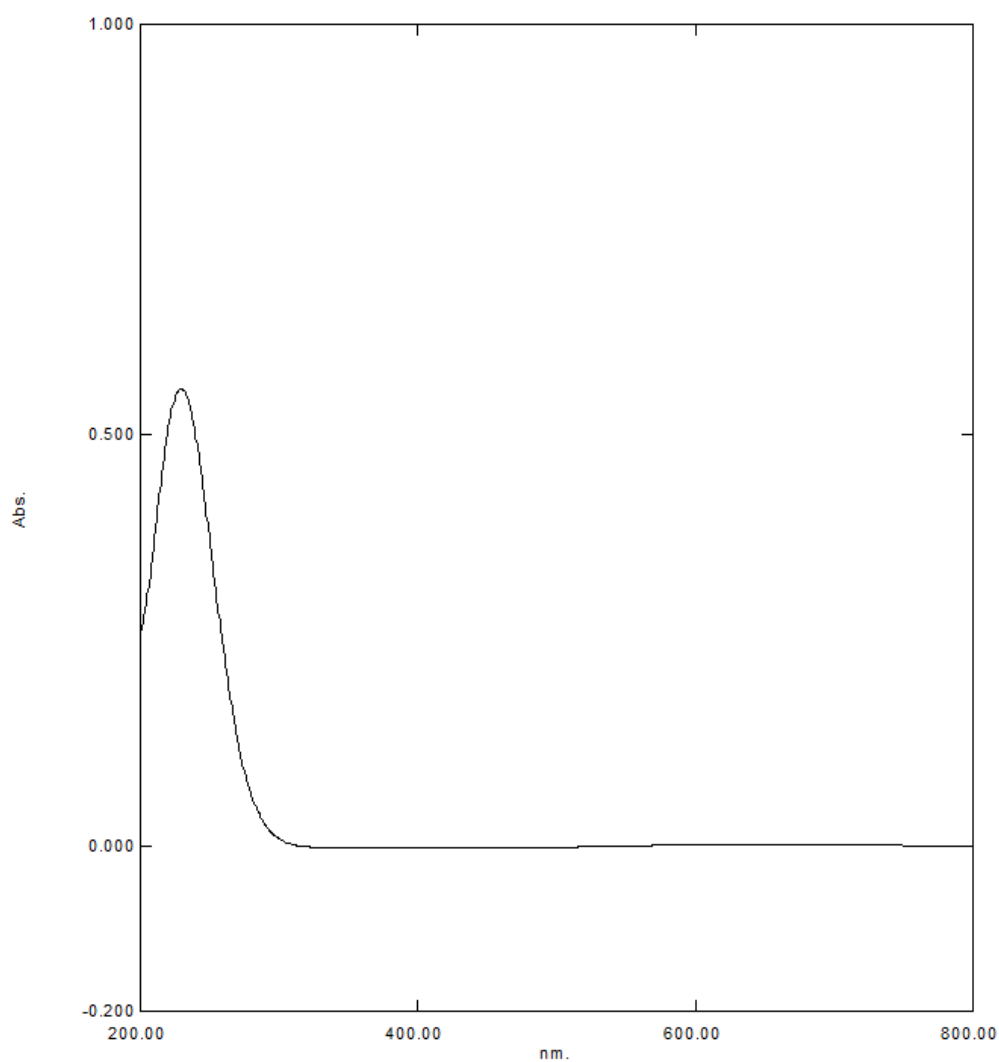
### RESULTS AND DISCUSSION

Synthesis of mannich amino methylation complex in a single step method. The synthesized aminomethylation derivatives were undergone characterization and the results.

mannich aminomethylation reaction is reaction involves complex formation of formaldehyde as a carbon acid. The compound confirmed by UV, IR spectroscopy. all the compound give the charteristic IR peak that proved that the presence of particular functional group.

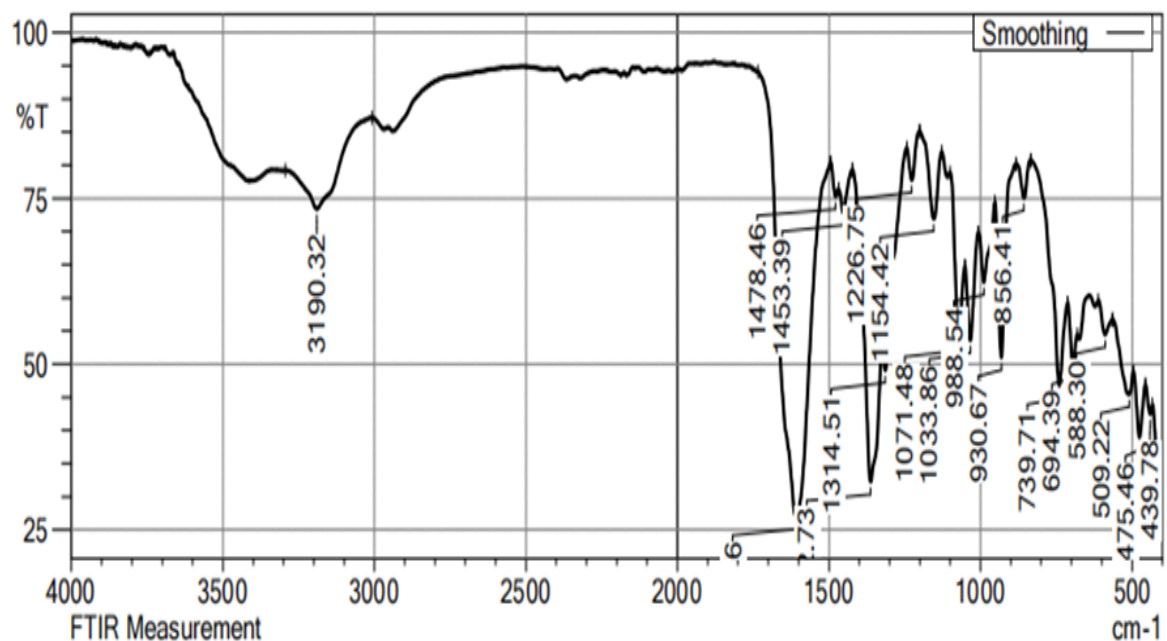
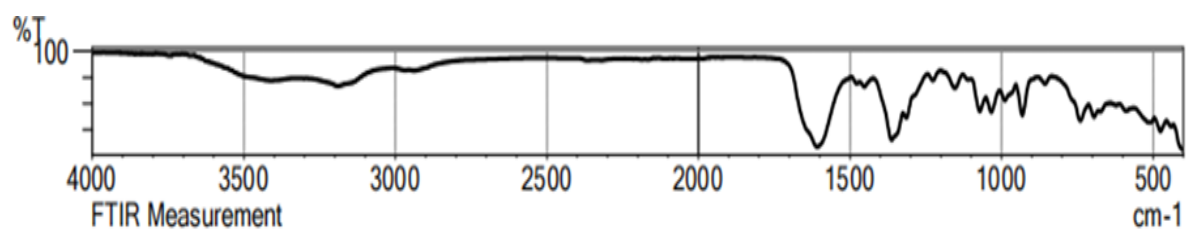
#### 5.1 UV SPECTROSCOPY RESLUT;

The UV spectroscopy the absorption spectrum of the compounds was taken in the region around 200-800nm using water solvent. The d-d transition peak for the complex is observed at 230nm. The mannich base product UV-visble spectrum shows in fig 1.



## 5.2 IR SPECTROSCOPY

IR spectrum of the sample was recorded using FTIR spectrometer. The IR spectrum of the compound or complex peaks at 439 ,475 ,509 ,588 ,694 ,739 ,856 ,930 ,988 ,1033,1154,1226,1314,1362,1453,1478,1608,3190. This based on the IR peaks the following structure has been assigned for the synthesized product.



|    | Peak    | Intensity | Corr. Intensity | Base (H) | Base (L) | Area     | Corr. Area | Comment |
|----|---------|-----------|-----------------|----------|----------|----------|------------|---------|
| 1  | 439.78  | 42.47     | 2.31            | 455.21   | 433.03   | 1241.148 | 28.968     |         |
| 2  | 475.46  | 39.00     | 8.82            | 495.71   | 455.21   | 2283.493 | 169.886    |         |
| 3  | 509.22  | 45.46     | 4.92            | 563.22   | 495.71   | 3384.784 | 194.368    |         |
| 4  | 588.30  | 54.44     | 3.69            | 611.45   | 565.15   | 2020.486 | 82.352     |         |
| 5  | 694.39  | 49.83     | 6.63            | 712.71   | 680.88   | 1477.623 | 103.426    |         |
| 6  | 739.71  | 46.80     | 17.19           | 834.23   | 712.71   | 4141.052 | 491.736    |         |
| 7  | 856.41  | 75.08     | 5.39            | 882.45   | 834.23   | 1050.019 | 107.163    |         |
| 8  | 930.67  | 51.04     | 25.17           | 952.85   | 882.45   | 2171.872 | 571.873    |         |
| 9  | 988.54  | 62.40     | 8.99            | 1006.86  | 952.85   | 1768.212 | 262.498    |         |
| 10 | 1033.86 | 53.60     | 13.16           | 1051.22  | 1006.86  | 1734.767 | 284.606    |         |
| 11 | 1071.48 | 54.01     | 16.58           | 1100.41  | 1051.22  | 1760.933 | 375.662    |         |
| 12 | 1154.42 | 71.97     | 11.19           | 1200.71  | 1127.41  | 1545.867 | 341.137    |         |
| 13 | 1226.75 | 77.78     | 5.75            | 1243.14  | 1200.71  | 794.414  | 107.879    |         |
| 14 | 1314.51 | 49.12     | 7.35            | 1325.12  | 1243.14  | 2741.898 | 83.243     |         |

|    |         |       |       |         |         |          |          |  |
|----|---------|-------|-------|---------|---------|----------|----------|--|
| 15 | 1362.73 | 32.26 | 30.66 | 1422.53 | 1325.12 | 4679.816 | 1364.167 |  |
| 16 | 1453.39 | 72.89 | 4.41  | 1467.85 | 1422.53 | 1090.245 | 85.872   |  |
| 17 | 1478.46 | 75.30 | 2.65  | 1494.86 | 1467.85 | 617.900  | 34.149   |  |
| 18 | 1608.66 | 27.04 | 59.91 | 1734.04 | 1494.86 | 9493.470 | 6451.139 |  |
| 19 | 3190.32 | 73.40 | 8.74  | 3294.47 | 3008.04 | 5796.701 | 988.971  |  |

## CHAPTER -6

### CONCLUSION;

Mannich amino methylation reaction involving copper glycinate complex. Using formaldehyde and diethyl succinate product in acidic medium. The synthesized compound by UV , IR spectroscopy.

The result obtained from obtained from this study conferment that the product has formed.

These product biological activity anticancer drug , antibacterial , antifungal , anticonvulsant , and antiviral activities in addition to several other bioactive , catalytic and detergent application.

Mannich base successfully synthesized a mannich aminomethylation as a carbon acid using formaldehyde and diethyl succinate.

## CHAPTER -7

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