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SYNTHESIS AND DELINEATION OF MODISH BETTI BASES VIA ORGANOSULPHUR COMPOUND

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ABSTRACT

The modish betti bases were conveniently synthesized in good yields from the three-component reaction of substituted phenol, aldehyde and thiourea without any other catalyst. In synthetic chemistry betti base is a key synthon. Furthermore, the synthesized compounds were confirmed by melting point and TLC. The structure of synthesized compounds was established by elemental analysis and various analytical techniques such as IR, 1HNMR and Mass spectral studies.

Keywords: Naphthol, Thiourea, Betti Mechanism, Synthon.

I. INTRODUCTION

At the beginning of the 20th century, Mario Betti discovered the three-component reaction of 2-naphthol, aryl aldehydes and ammonia or amines for the synthesis of aminobenzylnaphthols [1]. Now, this process has been known as the Betti reaction and the aminonaphthol product known as a Betti base[2]. The phenolic hydroxyl and amino groups in Betti bases can be as used as synthons. They have several biological applications, such as antibacterial, hypotensive activities[3-4]. Betti bases can be used as ligands to chelate with organometallic reagents in different reactions to provide highly efficient asymmetric reaction [5-6].

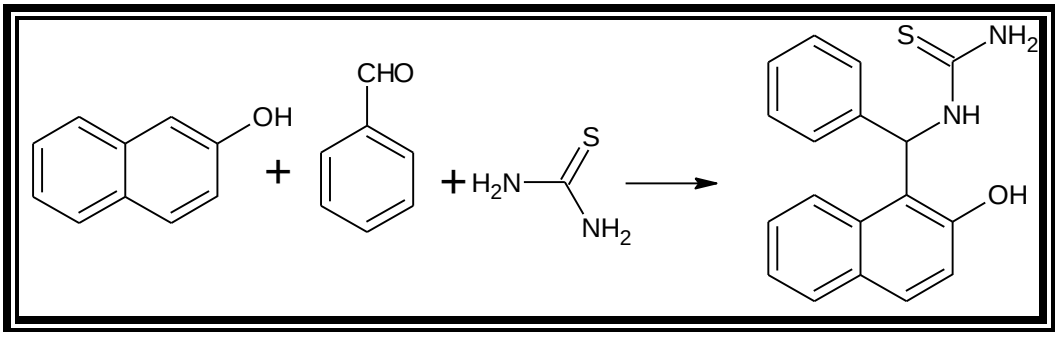
In the present paper, we report the synthesis and delineation of some modish betti bases. The structure of synthesized compounds were assigned based on Elemental analysis, I.R. 1H-NMR and Mass spectral data.

II. EXPERIMENTAL SECTION

Sigma-Aldrich and Merck chemicals were used as such without further purification. Solvents used for spectroscopic and other physical studies were reagent grade and were further purified by literature methods. Melting points were taken in open capillary tubes are uncorrected. IR spectra were obtained in on a Bruker spectrophotometer and expressed in wave numbers (cm^{-1}). 1H-NMR spectra were recorded on a Bruker Avance III 500 MHz spectrometer operating at 500 MHz for 1H-. The 1H-chemical shifts were expressed in ppm with reference to tetramethylsilane. Elemental analyses were performed at IISER Bhopal.

General procedure for synthesis of compounds: To a mixture of naphthol (2.0 mmol), aromatic aldehyde and ehanolic solution of organosulphur compunds were added slowly on hot water bath maintained at 90°C with constant stirring for 1 hr. the solid separated on cooling of reaction mixture was recrystallise by ethanol to give the pure product[7].

Synthesis of Betti base



Analytical and Spectral data of synthesized compounds

1-[(2-hydroxy naphthalen-1-yl)(phenyl) methyl] thiourea (compound- I):

Color: white, Yield: 63.36%, M.P: 158^0C, Elemental Analysis: C: 70.1%, H: 5.23%, N: 9.08 %, O: 5.19%, S: 10.40%, Molecular Formula: $C_{18}H_{16}N_2OS$.

IR (νmax, cm-1): 3755.87 (O-H streching), 3446.78 (primary amine, N-H streching), 3349.32 (Secondary amine, N-H sterching), 1137.17 (primary amine, C-N streching), 1215.91 (Secondary

amine, C-N sterching), 1079.77 (C-O), 683.34 (C=S). 1HNMR (CDCl3) δ: 9.03 (s, H, OH), 3.22 (s, 2H, NH2), 1.85 (d, H, NH), 6.55 (H, CH). MS:m/z:307.35.

1-[(5-amino-2-hydroxy phenyl) (phenyl) methyl] thiourea (compound- II):

Color: brown, Yield: 69.96% , M.P: 156^0C, Elemental Analysis: C: 61.51%, H: 5.53%, N: 15.37%, O: 5.85%, S: 11.73%, Molecular Formula: $C_{14}H_{15}N_3OS$ IR (νmax, cm-1): 3756.21 (O-H streching), 3371.27 (primary amine, N-H streching), 3243.15 (Secondary amine, N-H sterching), 1153.88 (primary amine, C-N streching), 1196.24 (Secondary amine, C-N sterching), 1077.06 (Phenol, C-O), 624.96 (C=S). 1HNMR (CDCl3) δ: 8.53 (s, H, OH), 2.35 (s, 2H, NH_2), 1.35 (d, H, NH), 7.02 (H, CH). MS:m/z: 273.35.

III. RESULTS AND DISCUSSION

Initially, the reaction conditions were examined using β-naphthol, aromatic aldehyde and thiourea as a model reaction. We found that, the reaction at room temperature proceeded much more slowly so we increase the temperature by hot water bath up to 90^0C at this temperature we good yield with low reaction time beyond this temperature the product become decompose, so we found 90^0C is appropriate temperature for the reactionto proceed.Under the optimized reaction conditions reactants smoothly give products 1-[(2-hydroxy naphthalen-1-yl)(phenyl) methyl] thiourea and 1-[(5-amino-2-hydroxy phenyl) (phenyl) methyl] thiourea in moderate yield.

In order to develop the scope of this reaction, other secondary amines, such as dimethylamine was also tested in the reaction. It is very disappointed to find that no expected Betti bases were formed. The structures of the prepared Betti bases were fully characterized with IR, HRMS and 1H NMR spectroscopy and the formulas of the selected compounds were confirmed by the elemental analysis.

IV. CONCLUSION

In summary, we investigated three-component reaction of β-naphthol or phenol with aromatic aldehyde and thiocarbamide and found the convenient synthetic protocol for the modish type of Betti bases. The potential uses of the reaction in synthetic and medicinal chemistry might be

quite significant. Further investigation with appropriate structural modification of the above compounds may result in therapeutically useful products.

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