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Synthesis of 1-(*3H*-imidazo[*4,5-b*]pyridin-2-yl)-2-(3,5-dimethyl-*1H*-pyrrol-2-yl)diazenes

Dayakar G.* and Jeyanthi A.[#]

**Department of Chemistry, Kakatiya University, Warangal, A.P.*
[#]Department of Chemistry, Satavahana University, Karimnagar, A.P.

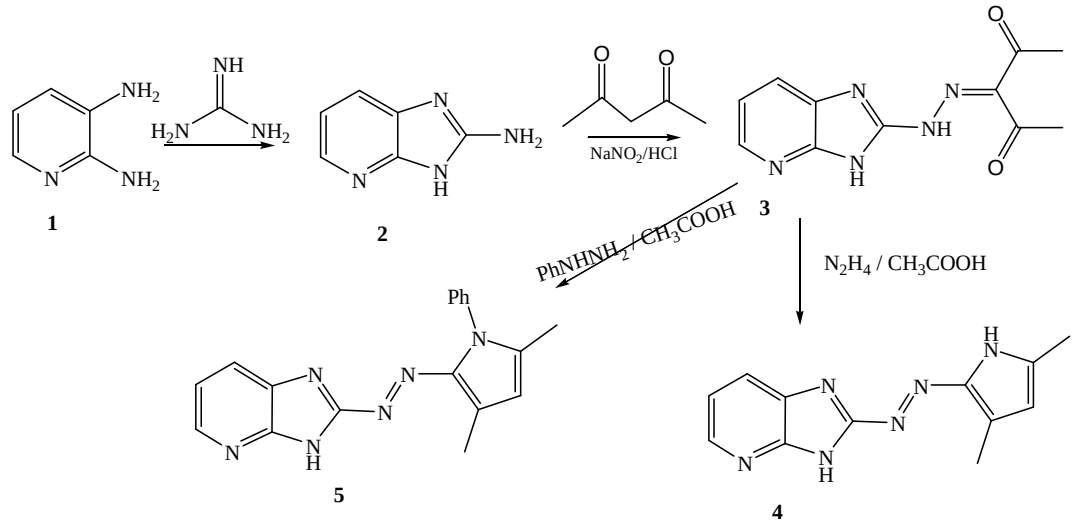
ABSTRACT

*3H-imidazo[4,5-b]pyridine-2-amine(**2**) was treated with acetyl acetone to form 1-(3H-imidazo[4,5-b]pyridine-2-yl)-2-(pentan-2,4-dione-2-ylidene)hydrazine(**3**). This was treated with hydrazine hydrate and phenyl hydrazine hydrate to give pyrrole derivatives 1-(3H-imidazo[4,5-b]pyridine-2-yl)-2-(3,5-dimethyl-1H-pyrrol-2-yl)diazenes(**4,5**).*

INTRODUCTION

The pyrrole derivatives were used as antimicrobial agents since several decades. For example,2-(2[I]-hydroxy benzoyl)pyrrole bromine derivatives have antimicrobial activity against *Staphylococcus aureus*, *Bacillus subtitles* and *Escherichia coli*. 2-methyl-1,3,5-trisubstituted pyrroles have significant activity. Tubercidin, toyocamycin and sangivamycin are naturally occurring pyrrolo(2,3-d)pyrimidine antibiotics having significant activity. Hence the below compounds were synthesized.

Scheme



MATERIALS AND METHODS

Chemicals and solvents were reagent grade and used without further purification. Melting points were determined on a capillary melting point apparatus and are uncorrected. The ^{1}H NMR was recorded in the indicated solvent on a Varian 500 MHz spectrometer with TMS as internal standard. All chemical shifts (δ) were reported in ppm from internal TMS. Mass spectra were measured on a Jeol JMS D-300 spectrometer. Infrared spectra were recorded in KBr on Brucher-IFS-66 FTIR spectrophotometer. The homogeneity of the compounds was checked using precoated TLC plates (E.Merk Kieselgel 60 F_{254}).

1-(3H-imidazo[4,5-b]pyridin-2-yl)-2-(pentan-2,4-dione -2-ylidene)hydrazine (3)
A cold mixture of acetyl acetone (0.01mole)and sodium acetate (0.01mole) in abs. ethanol (25ml) was added drop wise with stirring to solution of diazonium derivative of 3H-imidazo[4,5-b]pyridin-2-amine **(26)** over 10min, the stirring lasted for 30min, the reaction mixture was left about 2hrs. at room temp., red solid product then collected, with 86% yield. M.P.202^{o}C

IR: 3333 cm^{-1}(N-H), 2979 cm^{-1}(C-H aromatic), 1715 cm^{-1}(C=O), 1514 cm^{-1}(C=N); ^{1}H NMR (DMSO-d_6) : δ=2.21 (s, 6H), 7.35 (t, 1H), 7.85(d, 1H), 8.25 (d, 1H), 10.52 (brs, 1H), 13.01 (brs, 1H); Mass: m/z 245 (M+H).

1-(3H-imidazo[4,5-b]pyridin-2-yl)-2-(3,5-dimethyl-1H-pyrrol-2-yl)diazene (4)
A mixture of compound **(27)** (0.01mole) and hydrazine hydrate (0.02mole) was heated under reflux in acetic acid (25ml) for 10-12hrs, cooled and poured onto crushed ice and the solid product was obtained was filtered off with 82% yield. M.P.224^{o}C

IR: 3333 cm^{-1}(N-H), 2985 cm^{-1}(C-H aromatic), 1766 cm^{-1}(C=O), 1514 cm^{-1}(C=N). ; ^{1}H NMR (DMSO-d_6) : δ=2.21 (s, 3H), 2.41 (s, 3H), 6.14 (brs, 1H), 6.98 (t, 1H), 7.21 (t, 2H), 7.63 (dd, 1H), 7.81 (d, 1H), 7.94 (d, 2H), 8.12 (d, 1H), 13.02 (brs, 1H).; Mass: m/z 240 (M+H).

1-(3H-imidazo[4,5-b]pyridin-2-yl)-2-(3,5-dimethyl-1-phenyl-1H-pyrrol-2-yl)diazene (5)
A mixture of compound **(27)** (0.01mole) and phenyl hydrazine hydrate (0.02mole) was heated under reflux in acetic acid (25ml) for 10-12hrs, cooled and poured onto crushed ice and the solid product was obtained was filtered off with 91% yield. M.P.243^{o}C.

IR: 3333 cm^{-1}(N-H), 2932 cm^{-1}(C-H aromatic), 1715 cm^{-1}(C=O), 1514 cm^{-1}(C=N).; ^{1}H NMR (DMSO-d_6) : δ= 2.02 (s, 3H), 2.42 (s, 3H), 5.01 (brs, 1H), 7.25(t, 1H), 7.65(d, 1H), 7.98(d, 1H), 13.02(brs, 1H).; Mass: m/z 316 (M+H).

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