Synthesis and Characterization of 6-amino-4-(substitutedphenyl)-1-(2,4-dinitrophenyl)-3-methyl-pyrazolo[3,4-b]pyridine-5-carbonitrile

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ABSTRACT

6-amino-4-(substitutedphenyl)-1-(2,4-dinitrophenyl)-3-methyl-pyrazolo [3,4-b]pyridine-5-carbonitrile have been prepared by the refluxation for six hours of 4-(substitutedbenzylidene)-2-(2,4-dinitrophenyl)-5-methyl-2,4-dihydro-pyrazol-3-one, malononitrile and ammonium acetate in presence of ethanol. The intermediate 4-(substitutedbenzylidene)-2-(2,4-dinitrophenyl)-5-methyl-2,4-dihydro-pyrazol-3-one have been prepared by the refluxation for five hours of 2-(2,4-dinitro phenyl)-5-methyl-2,4-dihydro-pyrazol-3-one with substituted benzaldehyde in presence of glacial acetic acid. The synthesized compounds were characterized by means of their IR, ¹H-NMR spectral data and elemental analysis.

Introduction

Pyridine, a heterocyclic nucleus, played a pivotal role in the development of different medicinal agent and in the field of agrochemicals. In the recent past after considering the success of development of different medicinal agent and in the field of antimicrobial activity. Furthermore substituted derivatives of pyridines are associated with different biological properties like pesticidal, insecticidal and fungicidal activity. Furthermore substituted derivatives of pyrazolines exhibit antimicrobial activity. In view of these findings, it was contemplated to design and synthesize some new pyridine derivatives bearing pyrazolines and evaluate their antimicrobial activity.

Experimental

Melting points were taken in open capillary tube and were uncorrected. IR spectra were recorded on I.R. Spectrophotometer of Bruker scientific Model No. Alpha E and instrument used for NMR Spectroscopy was recorded in DMSO on Bruker Advance II 400 MHz spectrometer using TMS as an internal standard. Purity of the compounds were checked by tlc on silica- G plates.

Synthesis of 2,4-di-Nitro Phenyl Pyrazoline (SP-A)

For synthesis of SP-A, Mix together (0.4M) of redistilled Ethyl acetoacetate and (0.44M) of 2,4-di-Nitro Phenyl Hydrazine in a large evaporating dish. Heat the mixture on boiling water bath in the fume cupboard for about 2 hrs and stir from time to time with a glass rod. Allow the heavy reddish syrup to cool the somewhat, add about 100 ml of ether and stir the mixture vigorously. The syrup which is insoluble in ether, will solidify within 15 minutes. Filter the solid at the pump and wash it thoroughly with ether to remove the coloured impurities. Recrystallise it from hot water or from a mixture of Equal volume of ethanol and water. The yield of the product was 76% and the product melts at 95°C. Found: C(51.65%), H(3.08%), N(21.21%) IR; SP-A (cm⁻¹): 3079(=CH), 2912(-CH, Stretch), 1720(>C=O), 1600(=C=N Stretch), 1499(=C=N, aromatic ring), 1557(-N=N), 1463(-CH bending), 1343(-C-N<), 1245(-N-Nc), ¹H NMR (DMSO); SP-A: 2.55, singlate (3H) (-CH3), 2.30, singlate (2H)(-CH2-), 8.16-9.10, multiplate (3H) (Ar-H).

Preparation of 4-(substitutedbenzylidene)-2-(2,4-dinitrophenyl)-5-methyl-2,4-dihydro-pyrazol-3-one (SP-01-10)

A mixture of 2-(2,4-dinitrophenyl)-5-methyl-2,4-dihydro-pyrazol-3-one (0.01M) and substitutedbenzaldehyde (0.01M) in glacial acetic acid (25ml) was refluxed for 5 hours at a temperature of 120°C. The content was poured on to crushed ice. The isolated product was filtered, dried and crystallized from ethanol. IR; SP-01 (cm⁻¹): 3028(=CH), 2913(-CH, Stretch), 1676(>C=O), 1586(=C=N Stretch), 1487(=C=C, aromatic ring), 1564(-N=N), 1407(-CH3 bending), 1308(-C-N<), 1209(-N-Nc), 752(-C-Cl). ¹H NMR (DMSO); SP-08: 2.5871, singlate (3H)(-CH3), 2.4999, singlate (6H) [-N(CH3)]2, 7.7571, singlate (1H)(Ar-CH=, Vinylic), 7.3732-9.0397, multiplate (7H) (Ar-H).

Preparation of 6-amino-4-(substitutedphenyl)-1-(2,4-dinitrophenyl)-3-methyl-pyrazolo[3,4-b]pyridine-5-carbonitrile(Sp-11-20)

A mixture of 4-(substitutedbenzylidene)-2-(2,4-dinitrophenyl)-5-methyl-2,4-dihydro-pyrazol-3-one (0.01M) and malononitrile (0.01M) and ammonium acetate (0.08M) in absolute alcohol (30ml) and heated under refluxed for 6 hours. The content was pourer on to crushed ice. The product was isolated and crystallized from ethyl acetate. IR; SP-20 (cm⁻¹): 3344(>NH-), 3063(=C=H), 2918(-CH3, stre), 2192(<C=N), 1574(>C=N, stre), 1524(>C=C, aromatic ring), 1495(=N-O), 1414(-CH=, bend), 1397(-C-N<), 1273(-N-Nc).

¹H NMR (DMSO); SP-16: 2.5899, singlate (3H)(-CH3), 3.7360, singlate (3H)(-OCH3), 4.1728, singlate (2H)(-NH2),

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References