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Synthesis and characterization of some newly synthesized Nitro and Bromo substituted 3, 5-diaryl isoxazolines

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ABSTRACTS
Five membered heterocycles like isoxazolines have found wide application as pharmaceutical and agrochemical agents. The present work deals with the synthesis of bromo and nitro substituted 3, 5-diarylisoxazolines from bromo and nitro substituted chalcone and hydroxylamine hydrochloride in the presence of ethanol solvent by refluxing for 2 hours.

INTRODUCTION:

The azole containing one oxygen and one nitrogen atom at 1, 2-position are designated as isoxazoles.

The dihydroderivative of isoxazole are known isoxazolines. A heterocyclic compound is one which possesses a cyclic structure with at least two different kinds of atoms in the ring. Compound incorporating heterocyclic ring systems continue to attract considerable interest due to the wide range of biological activities. Amonst them five membered heterocyclic compounds occupy a unique place in the realm of natural and synthetic organic chemistry. Five membered heterocycles like isoxazolines have found wide application as pharmaceutical and agrochemical agents.

The synthesized 3,3-diaryl-4-aroyl isozolines from 3-aroy flavnones and 3-aroyl chomones by the action of hydroxylamine hydrochloride in ethanol[1] also the synthesis of 3-(2'-hydroxy-3'-bromo-5'-5'-ethylchalcone and hydroxylamine hydrochloride in ethanol[2] and the preparation of substituted fluro isoxazolines from flurochalcones in DMF[3] also the preparation of of some chlorosubstituted nitrogen and oygen containing heterocycles and their use in controlling horticulture crops pathogens.[4]The Clean and effeIcient synthesis of isoxazalose derivative in aquaes media Synthesis of biocyclic isoxazoles isozaxoliness via intra molecular nitrile oxidecycloaddition[5-6]. The Synthesis and characteraisation of some novel isoxazoles via chalcone intramediates and The novel isoxazolines ectoparasiticide furalaner selective inhibition of orthopad aminobutyric acid and l-glutamate gated chloride and chanels and insteticidals acaricidal activity.[7-8] Expeditious preparation of isoxazole from isoxazoline and advanced intermediate for functional materials and Synthesis of some 3,5-diary-2-isoxazolines derivatives in ionic media[9-10]

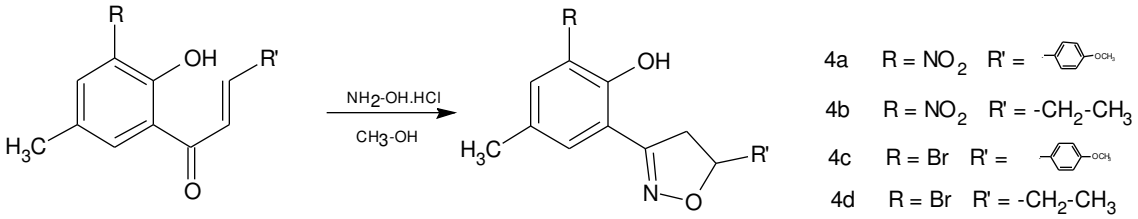
EXPERIMENTAL WORK:

The interaction of hydroxyl amine hydrochloride and chalcones has the most convenient method for the synthesis of isoxazolines.

The synthesis of isoxazoline from 1-(2-hydroxy-3-nitro-5-chlorophenyl)-3-(4-chloro phenyl) chalcone (0.01mole) treated with ortho-chlorobenzaldehyde in presence of KOH. The melting points of these compounds were recorded on 'Tempo' melting point apparatus and are uncorrected. The carbon nitrogen hydrogen and oxygen analysis was carried out on 'Carlo Ebra 1106' analyzer. The IR spectra were recorded on 'Perkin-Elmer' Infra Red spectrophotometer. The PMR spectra recorded on DRX300 spectometer in $CDCl_3$. Purity of this compound tested by TLC.

A) preparation of 3-(2-hydroxy-3-bromo/nitro-5-chlorophenyl)-5-(4 methoxychloro-phenyl)-isoxazoline (IIa/b)

A mixture of 1-(2-hydroxy-3-bromo/nitro-5-chlorophenyl)-3-(4-chloro phenyl)- chalcone(0.01 mole) and hydroxyl amine hydrochloride (0.02 mole)was refluxed for 2.5 hr.in methanol (25ml).The reaction mixture was decomposed crystalline solid product thus separate was filtered and crystallized from ethanol.



3 (a-d) 4 (a-d)

SPECTRAL INTERPRETATION:

Compound (4b): $C_{12}H_{14}N_2O_4$; **IR (cm^{-1}):** 3398.57 (-OH bonding), 3072.60 (Aro. stret.), 2927.94 (sp^3 C-H stret.), 1448.54 (-CH_2 bend.), 1365.60 (-CH_3 bend.), 1537.27 (NO_2 stret.), 1200 (C-O stret.), **NMR (δ ppm):** 12.86 (s, 1H, Ar-OH), 7.5-7.6 (m, 2H, Ar-H), 2.5 (d, 2H, Hetero –CH_2), 6.7 (m, 1H, Hetero –CH), 2.24 (m, 2H, -CH_2), 2.2 (t, 3H, -CH_3).

Compound (4d): $C_{12}H_{14}BrNO_2$; **IR (cm^{-1}):** 3435 (-OH bonding), 3055.24 (Aro. stret.), 2995.45 (sp^3 C-H stret.), 1319.31 (-CH_3 bend.), 578.64 (C-Br stret.), 1400.32 (-CH_2 bend.), 1319 (C-O stret.), **NMR (δ ppm):** 11.8 (s, 1H, Ar-OH), 6.8 (m, 2H, Ar-H), 2.27 (s, 3H, Ar-CH_3), 2.5 (d, 2H, Hetero –CH_2), 6.7 (m, 1H, Hetero –CH), 2.24 (m, 2H, -CH_2), 2.2 (t, 3H, -CH_3).

RESULT AND DISCUSSION

Synthesis of some Nitro and Bromo analogues of 3, 5-Diaryl Isoxazolines were achieved and their structures were confirmed on the basis of UV, IR and NMR spectral analysis.

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