Synthesis And Characterisation of newly Synthesized Isoxazoline

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Abstract:

A new series of bromo and nitro substituted isoxazolines (3a-d) were synthesized by reacting bromo and nitro substituted chalcones (2a-d) with hydroxyl amine hydrochloride respectively. All these compounds were characterized by means of their UV,IR,^{1}H NMR, spectroscopic data and elemental analysis.

Keywords: Chalcones,Isoxazolines

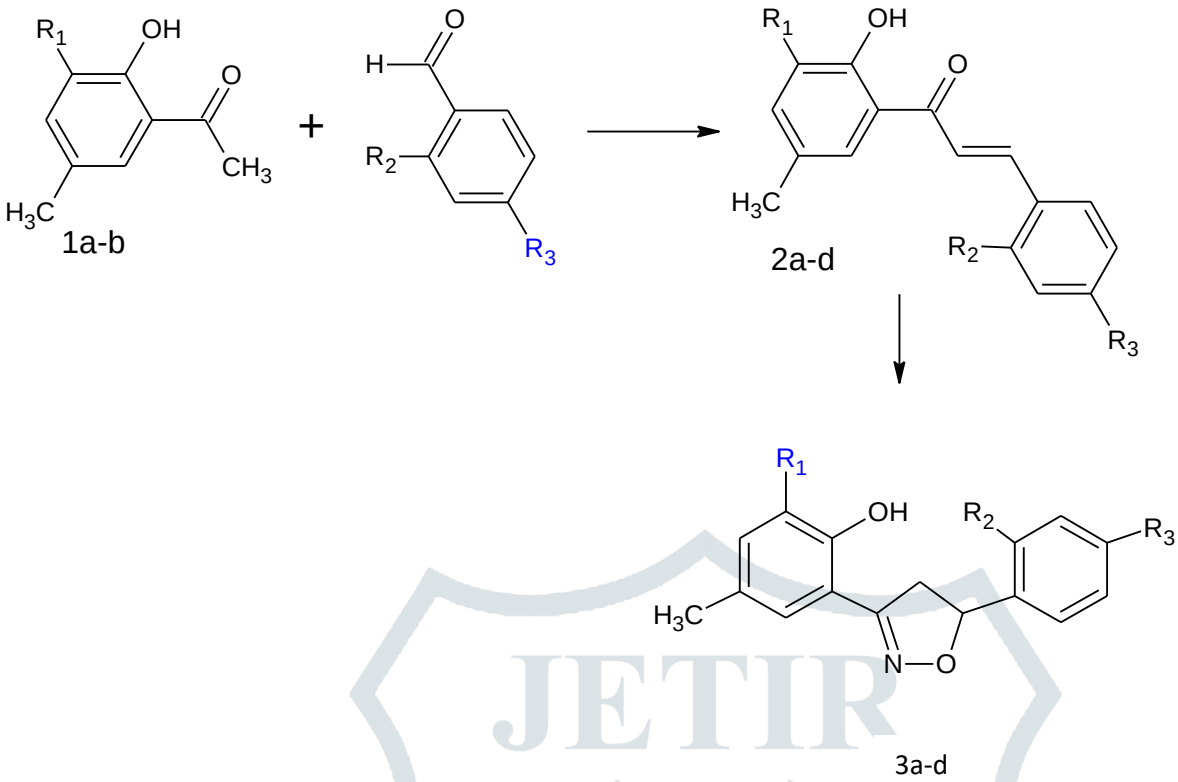
INTRODUCTION:

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

Experimental

General procedure for synthesis of chalcones (2a-d)

A mixture of substituted acetophenones (0.01 mole) and substituted benzaldehyde (0.01 mole) was stirred in ethanol (15 ml) and to this solution 10% potassium hydroxide added gradually with constant stirring. The reaction mixture was kept overnight at room temperature and then it was poured into crushed ice and acidified with dil. HCI. The product precipitates out was filtered washed with $NaHCO_3$ solution and crystallized from ethanol Scheme 1



General procedure for synthesis of isoxazolines (3a-d)

Bromo and nitro substituted chalcone (2a-d) (0.01 mol) and hydroxyl amine hydrochloride (0.02mol) was refluxed in ethanol (20 ml) and piperidine (0.5ml) for about 1.5 hrs. After cooling; the reaction mixture was acidified with HCl. The solid product thus separated was filtered, washed first with sodium bicarbonate solution (10%) and then with water. Finally it was crystallized from ethanol to get isoxazolines (3a-d). Physical and analytical data of compounds are given in table 1.

Physical and analytical data of compounds (2a-d,3a-d).

table 1.

Compound no.	R_1	R_2	R_3	M.F.	m.p.^{0}C	Yield %	C %	H %	N %
2a	Br	H	Cl	$C_{16}H_{12}O_2BrCl$	101	72	53.98	3.01	--
2b	NO_2	H	Cl	$C_{16}H_{12}O_4NCl$	115	68	59.95	3.01	4.01
2c	Br	Cl	Cl	$C_{16}H_{11}O_2BrCl_2$	82	72	49.01	2.03	--
2d	NO_2	Cl	Cl	$C_{16}H_{11}O_4NCl_2$	102	78	54.02	2.99	3.10
3a	Br	H	Cl	$C_{16}H_{13}BrClNO_2$.	120	60	52.01	3.07	3.00
3b	NO_2	H	Cl	$C_{16}H_{13}ClN_2O_4$.	135	76	56.9	3.02	8.00

3c	Br	Cl	Cl	$C_{16}H_{12}BrNO_2Cl_2$.	160	70	47.00	2.03	3.02
3d	NO_2	Cl	Cl	$C_{16}H_{12}Cl_2N_2O_4$.	145	80	51.90	3.02	7.01

Results and Discussion

The structure of synthesized compounds (2a-d,3a-d) were confirmed on the basis of spectral and elemental analysis .The IR spectrum of **2a-d** exhibited a band due to –OH str.(3350.88cm^{-1}), >C=O str. (1691.59 cm^{-1}),-C=C-str.(1613.84 cm^{-1}), -Cl str.(1135.52 cm^{-1}),-Br str.(1059cm^{-1}).Futher,in^{1}H NMR spectrum, the appearance of a signal at δ 2.4 (s,3H,-CH3), 7.09 to 7.83(m,6H, Ar–H), 7.85(d,1H,-COCH=CH-),8.2(d,1H,-COCH=CH-),11.17(s,1H,-OH)confirms the presence of chalcone.

Similarly ,the structure of compounds **3a-d** was confirmed on the basis of spectral and elemental analysis.The IR spectrum of **3a-d** exhibited a band due to H-bonded O–H stretching (3241.21cm^{-1}), aromatic stretching, >C–H (3087.43 cm^{-1}), Aliphatic C–H stretching 2922.35, –C=N- str.(1582.55cm^{-1}), >C–O stretching (1288.00 cm^{-1}) and -C–Br stretching (1102.97 cm^{-1}). Further, in their in^{1}H NMR spectrum, the appearance of a signal at δ 2.33 (s,3H,Ar-CH_3); δ 3.5 (d, 2H, Heterocyclic –CH_2–CH–) , δ 5.06 (s,1H,-Ar–OH), δ 6.8 (t, 1H, Heterocyclic –CH2–CH–), δ 7.09-7.54 (m,6H,Ar–H) δ 1.2 (s,1H,-CH), δ 2.5(s,1H,-NH), δ 2.3.3 (s,3H,-CH3), δ 6.9(d,1H, =C–H), δ 7.3 to 8.3(m,11H, Ar–H), and δ 12.3(s,1H,-OH) confirms the presence isoxazoline.

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