Asian Journal of Chemistry Vol. 10, No. 3 (1998), 597–599

NOTE

Synthesis of Some New 1-Carboxamido-3-(substitued -2-hydroxy phenyl)-5-aryl-Δ^2-pyrazolines

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2′-Hydroxychalcone an aqueous solution of semicarbazide hydrochloride were refluxed in ethanol for 3 h. The reaction mixture was cooled, filtered and crystallised from ethanol-acetic acid mixture to get 1-carboxamido-3-(substituted-2-hydroxyphenyl)-5-aryl-Δ^2-pyrazoline. All these synthesised new pyrazolines were tested against organism. The Minimum Inhibitory Concentration (MIC) values were determind by using serial dilution method.

Tetrolic aldehyde or its acetal was reported to give 5-methyl pyrazole-1-carboxamide by shaking the semicarbazone with alkali for two days.[1] Hashash *et al.*[2] synthesised 1-carboxamido-pyrazolines from epoxyketone and semicarbazide. 3 or 5 Phenyl pyrazole-1-carboxyamido synthesised[3] when semicarbazide reacts with hydroxymethylene acetophenone. 2′-Hydroxychalcones on treatment with simicarbazide hydrochloride in ethanol and in DMF[4] gives 1-carboxamido-3-(2-hydroxyphenyl)-5-aryl-Δ^2-pyrazolines. Literature survey reveals that 1-carboxamido-pyrazoline was not synthesised from chloro, chlorobromo, chroronitro substitued chalcone. Hence it was thought interesting to prepare some new 1-carboxamido-pyrazoline.

2′-Hydroxy-5′-chlorochalcone (Ia) (0.01 mole) and aqueous solution of semicarbazide hydrochloride (0.02 mole in 2 mL H_2O) were refluxed in ethanol (30 mL) for 3 h. The product thus obtained on cooling the reaction mixture was filtered and crystallised from ethanol-acetic acid mixture to get 1-carboxamido-3-(2-hydroxy-5-chlorophenyl)-5-phenyl-Δ^2-pyrazoline (IIa), m.p. 220°C, yield 80%.

Properties of Compound (IIa)

1. The compound(IIa) is yellow crystalline campound m.p. 220°C.
2. It gives green colouration with ethonolic $FeCl_3$ solution indicating the presence of free —OH group.

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3. It developed deep blue colouration with conc. H_2SO_4 showing the presence of —C(=O)—CH=CH— linkage,

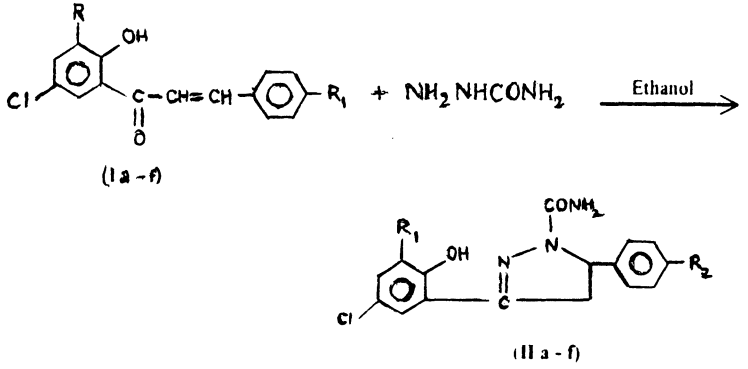
4. It remains unchanged on boiling in acetic acid.

5. The analytical results indicate the molecular formula of the compound as $C_{16}H_{14}O_2N_3Cl$.

6. The main absorption bands observed in IR spectum of (IIa) can be correlated as IR 3400 (bonded OH), 3280–3220 ν(—$CONH_2$) and 1670 cm^{-1} ν(C=N of pyrazoline).

7. NMR of compound (IIa) recorded in $CDCl_3$: 2.06 δ (s, Residual portion of acetic acid), 274 (dd, 1H, >CH), 3.16 (dd, 1H, > CH_3H), 5.15 (dd, 1H, >CH_x—), 5.4–6.1 (s, 2H, $CONH_2$), 6.9–7.8δ (m, 8H, Ar—H) and 9.48 (s, 1H, —OH).

From the above properties compound (IIa) was assigned the structure as 1-carboxamido-3-(2-hydroxy-5-chlorophenyl)-5-phenyl-Δ^2-pyrazoline.



The other pyrazolines prepared are listed in Table-1.

TABLE-1
PHYSICAL PROPERTIES OR PYRAZOLINES

Comp.	R_1	R_2	m.p. (°C)	Yield %	Molecular formula	m.w.
IIa	H	H	220	80	$C_{16}H_{14}O_2N_3Cl$	315.5
IIb	H	OCH_3	222	75	$C_{17}H_{16}O_3N_3Cl$	329.5
IIc	Br	H	226	78	$C_{16}H_{13}O_2N_3ClBr$	393.5
IId	Br	OCH_3	218	75	$C_{17}H_{15}O_3N_3ClBr$	407.5
IIe	NO_2	H	218	79	$C_{16}H_{13}O_4N_4Cl$	360.5
IIf	NO_2	OCH_3	214	73	$C_{17}H_{15}O_5N_4Cl$	374.5

Antimicrobial activity

Antimicrobial activity of pyrazolines (IIa–f) were assayed against the test

organism *Staphylococcus aureus*, *Streptococcus Pyogenes*, *Streptococcus agalactiae*, *Streptoccocus faecalis*, *Corynebacterium ulcerans*, *Corynebacterium minuttissimum*, *Clostridium septicum*, *Clostridium tetari* and *Escherichia coli*.

The MIC values of the compound were determined by serial dilution method.[5, 6] From Table-2 it is seen that pyrazolines (IId) and (IIe) are more active as compared to other pyrazolines due to the presence of nitro and bromo groups respectively in the structure.

TABLE-2
THE MINIMUM INHIBITORY CONCENTRATION OF COMPOUNDS (IIa–f) (values in %)

Compd.	*S. aureus*	*S. pyogenes*	*S. agalactiae*	*S. faecalis*	*C. ulcerans*	*C. minutti-ssimum*	*C. septicum*	*C. tetari*	*E. coli*
IIa	0.44	0.20	0.39	0.26	0.11	0.12	0.14	0.12	0.11
IIb	0.12	0.19	0.24	0.29	0.23	0.17	0.14	0.29	0.40
IIc	0.44	0.53	0.42	0.51	0.49	0.21	0.20	0.09	0.10
IId	0.12	0.12	0.12	0.10	0.11	0.09	0.03	0.98	0.12
IIe	0.04	0.05	0.11	0.15	0.17	0.29	0.52	0.54	0.11
IIf	0.52	0.44	0.42	0.48	0.32	0.40	0.39	0.52	0.42

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(*Received:* 18 Septamber 1997; *Accepted:* 17 February 1998) AJC-1446