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SYNTHESIS OF SOME NEW SCHIFF BASE OF IODOVANILLIN WITH POSSIBLE FUNGICIDAL ACTIVITY

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Abstract

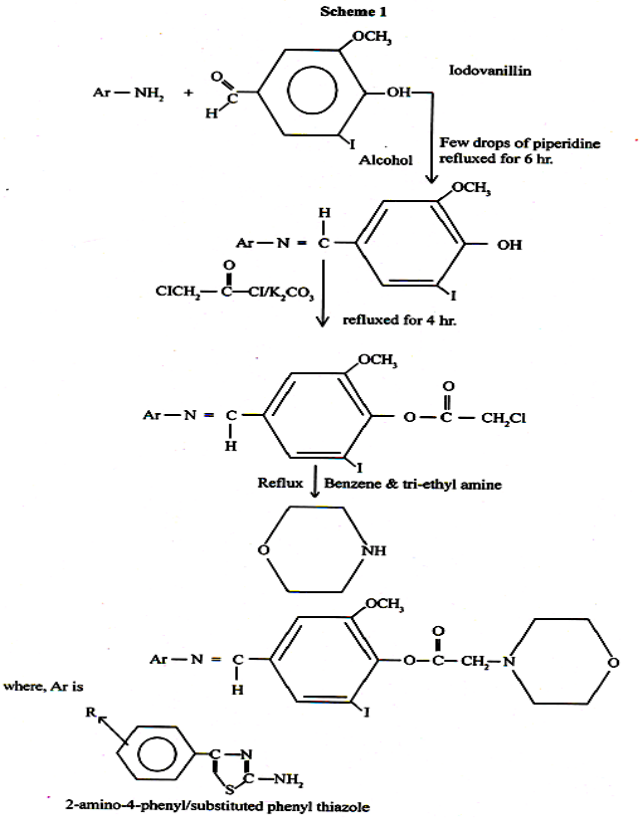
A new series of schiff bases derived from 2-amino-4-phenyl/substituted phenyl Thiazole and Iodovanillin were synthesized which on condensation with chloroacetyl chloride and subsequent reaction with morpholine yield corresponding acetoxy derivatives. The compounds were characterized by elemental analysis and IR and NMR spectra. They were also screened for their fungicide activity.

Keywords: 2-amino-4-phenyl; Schiff bases; Morpholine.

Introduction

Schiff bases are well known to have pronounced biological activities. Their ready synthesis and myriad properties have contributed greatly to their popularity and to the study of many biological systems. Schiff base complexes have been suggested as models for enzymes such as galactose oxidase. Schiff bases and their metal complexes derived from thiazole amines have been reported. In the present investigation, the synthesis of a new series of Schiff bases derived from 2-amino-4-phenyl/substituted phenyl thiazoles and Iodo vanillin has been undertaken. Condensation of the Schiff bases with chloroaceyl chloride and subsequent reaction with morpholine yielded corresponding morpholino acetoxy derivatives. The steps involved in the synthesis are sketched in Scheme 1. 2-amino-4-phenyl/substituted phenyl thiazole were prepared by the method of Dodson & King.

The compounds have been characterised by their sharp m.p., elemental analysis and ir spectra and nmr spectra. In literature various



synthetic antifungal compounds have been reported but the condensation 2-amino-4-pheny/substituted phenyl thiazole with Iodovanillin have not been reported, consequently their antifungal activity reported may open a new knowledge in antifungal compounds.

Experimental

Melting points were determined in open capillary tubes and are uncorrected. Infrared and nmr spectra is recorded.

[I] Synthesis of N-[4-Phenyl-2-thiazolyl-2-imino-[4'hydroxy-3'-iodo-5'-methoxy] benzal imine :-

To a mixture of iodovanillin (0.001 mol) and 2-Amino-4-phenyl thiazole dissolved in ethanol (25 ml) and piperidine (3-4 drops) was added and the mixture was refluxed for 6 hrs. and the separated solid were crystallised from ethanol yield 55% and m.pt.-133^{0}C.

[II] Synthesis of N-[4-phenyl-2-thiazolyl]-2-imino-(4'-chloroacetoxy-3'-iodo-5'-methoxy) benzal imine :-

To the compound [I] (0.01 mole) add drop wise chloroacetyl chloride in K_2CO_3 using benzene as a solvent and refluxed on water bath for 4 hours. The solid which separated on cooling was filtered and recrystallized from ethanol yield- 50% and m.pt. - 120^{0}C.

[III] Synthesis of N-[4-phenyl-2-thiazolyl]-2-imino-(3'-Iodo-5'-methoxy-4'-morpholinoacetoxy) benzal imine :-

To the compound (0.004 mole) add morpholine (0.01 mole) in dry benzene (25 ml) was refluxed on water bath for 8 hours. Excess benzene was removed under reduced pressure. The solid mass obtained was filtered, dried and recrystallised from ethanol.

Yield = 60%

M.P. = 125^{0}C

Similarly, N-[4-(p-subst.)-phenyl-2'-thiazolyl-2-imino-(3'-Iodo-5'-methoxy-4'-morpholino acetoxy) benzalimine were synthesized using similar procedure and their analytical data are incorporated in the following table (1).

IR spectra

The ir spectra of Schiff bases showed a broad medium band around 3100 cm^{-1} (bonded OH). Disappearance of the OH band and appearance of a new band around 1715-1725 cm^{-1} established the formation of the acetoxy derivatives. The characteristic absorption peaks is the region of 1645-1620 cm^{-1} and 1250 cm^{-1} corresponds to (C-N) and (C-N) bond established the formation of morpholino derivative.

Compound nmr

1. 1. 7.1-7.2 (5H, m, ArH), 7.0(1H, s, -CH) thiazole ring, 7.5(1H, s, -N=CH), 6.7-7.2(2H, m, ArH), 3.73, (3H, t, -OCH_3), 2.27 (2H, d, -CH_2), 2.6-3.6, (8H, m, morpholine - CH_2), 1.09-1.1 (1H, -NH in morpholine)
2. 2. 7.1-7.2(4H, m, ArH), 7.0 (1H, s, -CH) thiazole ring, 7.5(1H, s, -N=CH), 6.7-7.2 (2H, m, ArH), 3.73 (3H, t, -OCH_3), 2.27 (2H, d, -CH_2), 2.6 - 3.6 (8H, m, morpholine -CH_2), 1.09-1.1 (1H, -NH in morpholine)
3. 3. 6.8 - 7.3 (4H, m, ArH), 7.0 (1H, s- Cu) thiazole ring, 7.5 (1H, s, -N = CH), 6.7 - 7.2 (2H, m, ArH), 3.73 (3H, t, -OCH_3), 2.27 (2H, d, -CH_2), 2.6- 3.6, (8H, m, morpholine), 1.09-1.1 (1H, - NH Ch Morpholine)
4. 7.0-7.5 (4H, m, ArH), 7.0 (1H, s-CH) thiazole ring, 7.5 (1H, s, -N = CH), 6.7 - 7.2 (2H, m, ArH), 3.73 (3H, t, -OCH_3), 2.27 (2H, d, -CH_2), 2.6- 3.6, (8H, m, morpholine), 1.09- 1.1 (1H, -NH in morpholine)
5. 6.6-7.2 (4H, m , ArH), 7.0 (1H, s, -CH) thiazole ring, 7.5 (1H, s, -N = CH), 6.7 - 7.2 (2H, m, ArH), 3.73, (3H, t, -OCH_3), 2.27 (2H, d, -CH_2), 2.6 - 3.6 (8H, m, morpholine - CH_2), 1.09-1.1 (1H, -NH in morpholine)
6. 7.4-8.2 (4H, m, ArH), 7.0 (1H, s, -CH) thiazole ring, 7.5 (1H, s, - N= CH), 6.7 - 7.2 (2H, m, ArH), 3.73 (3H, t, -OCH3), 2.27 (2H, d, -CH_2), 2.6- 3.6 (8H, m, morpholine - CH_2), 1.09 - 1.1 (1H, -NH in morpholine)
7. 7. 6.6 - 7.2 (4H, m, ArH), 5.0 (1H, s, -OH) in Aromatic ring, 7.0 (1H, s, -CH) thiazole ring, 7.5 (1H, s, -N=CH), 6.7 - 7.2 (2H, m, ArH), 3.73 (3H, t, -OCH_3),

2.27 (2H, d, -CH_2), 2.6- 3.6 (8H, m, morpholine -CH_2), 1.09 - 1.1 (1H, -NH in morpholine)

8. 7.0- 7.2 (4H, m, ArH), 2.35 (3H, t, -CH_3) in Aromatic ring, 7.0 (1H, s, -CH) thiazole ring, 7.5 (1H, s, -N=CH), 6.7- 7.2 (2H, m, ArH), 3.73 (3H, t - OCH_3), 2.27 (2H, d, -CH_2), 2.6 - 3.6 (8H, m, morpholine CH_2), 1.09 - 1.1 (1H, -NH in morpholine)
9. 6.6-7.2(4H, m, ArH), 3.98 (2H, d, -OCH_2 gp) in Aromatic ring, 1.35 (3H, t, -CH_3 gp), 7.0 (1H, s, -CH) thiazole ring, 7.5 (1H, s, -N =CH), 6.7-7.2(2H, m, Ar H), 3.73 (3H, t, -OCH_3), 2.27 (2H, d, -CH_2), 2.6-3.6(8H, m, morpholine –CH_2), 1.09-1.1(1H, -NH in morpholine)

Antifungal Screening

From time to time, different methods, were used by different worker's. In the present studies. The anti-fungal screening of the compounds was under taken as given by schmitz and Alexopoulous[22-23] (1930).

A few benzalimines were evaluated for fungicidal activity by food and poison the following fungus are taken

1. Alternaria alternata 2. Curvularia lunata
3. Fusarium solani

Effect of newly synthesised anti-fungal compounds-against these fungus at optimum temperature of 28 $\pm$ 1^0C (After 7 days incubation) was observed.

Results and Discussion

Characteristics of compounds (III) and results of antifungal activity are given in table (I).

It is evident from fungal screening data that all the newly synthesized compouns tested were found satisfactorily, superior antifungal (Baristin) compounds. Mostly synthesized compound showed inhibition of the fungal growth in nitro test.

Table – 1

S. N.	Nature of Ar.	M.P. OC	Elemental Analysis				% Inhibition of		
			% of N		% of S		Allernoria Allernata	Fusarium	Curevularis Lunata
			C	F	C	F			
1	2-Amino-4-phenyl thiazole	141	7.44	7.40	5.67	5.65	95.23	95.23	93.09
2	2-Amino-4-(p-chloro) phenyl thiazole	143	7.01	7.00	5.34	5.32	95.55	95.55	91.94
3.	2-Amino-4-(p-Fluoro) phenyl thiazole	148	7.21	7.40	5.49	5.48	96.05	96.05	91.07
4.	2-Amino-4-(p-Bromo) phenyl thiazole	145	6.54	6.51	4.98	4.95	96.21	95.23	96.05
5.	2-Amino-4-(p-methoxy) phenyl thiazole	152	7.43	7.40	5.66	5.64	96.87	94.40	94.73
6.	2-Amino-4-(p-nitro) phenyl thiazole	158	9.19	9.15	5.25	5.20	95.39	92.26	93.09
7.	2-Amino-4-(p-hydroxy) phenyl thiazole	159	7.44	7.40	5.67	5.65	94.90	92.76	92.59
8.	2-Amino-4-(p-methyl) phenyl thiazole	175	7.01	7.00	5.34	5.32	95.23	94.90	96.21
9.	2-Amino-4-(p-ethoxy) phenyl thiazole	165	7.21	7.40	5.49	5.48	97.03	96.05	96.87

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