Procedure International Journal of Science and Technology

(International Open Access, Peer-reviewed & Refereed Journal)

(Multidisciplinary, Monthly, Multilanguage)

ISSN : 2584-2617 (Online)

Volume- 1, Issue- 8, August 2024

Website- www.pijst.com DOI- https://doi.org/10.62796/pijst.2024v1i8003

Synthesis of Some New Schiff bases with Possible Fungicidal Activity

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A new series of schiff's bases were synthesised by the condensation of cycloadditition of schiff bases with chloroacetyl chloride and triethylamine in dioxane to afford the corresponding azetidinones, similarly various azitidinones were synthesised. The compounds were characterised by elemental analysis, IR and NMR spectra and were tested for their fungicidal activities. Schiff bases possess diversified biological applications, various 2-azetidinones shows a variety of pharmacological and microbiological activities. Morever compounds containing 2-azetidinones ring system are shown to possess market biological activities1 particularly Fungicidal activity2,3. The present paper reports the synthesis of some new bases 2-azetidinones4. The Schiff bases on reaction with chloroenetyl chloride in dioxane in the presence of triethyl amine yielded 2-azetidinones.

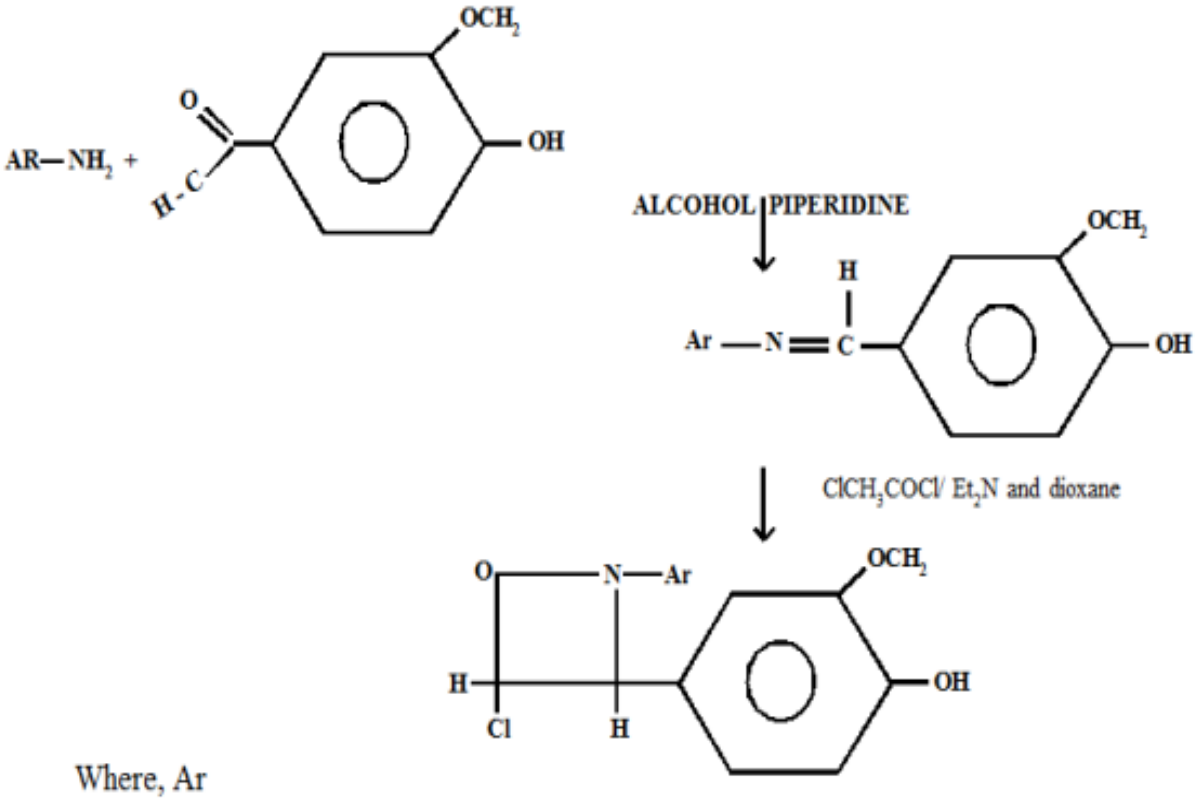
Experimental

The synthesised compound have been characterised on the basis of their elemental analysis, IR spectra and NMR spectra. The purity of the compound has been checked by thin layer chromatography.

2-Amino-5-chloro-4-phenyl thiazole (2g) was treated with conc. HCL (3ml) and the hydrochloride5 salt was dissolved in 25 ml of glaial acetic acid. The mixture was stirred well, chlorine gas was passed. Successively and stready rate high this solution labies in another flask containing hydrite salt solution of thiazole6 was shaken occasionally. The residue was treated with water and was basified with liquid ammonia. The free base librated was seprated by filteration and crystallized from ethanol.

N-[5-Choloro-4-Phenyl-2-Thiazolyl]-2- imino-[3-Methoxy-4-hydroxy]-benzylidene-

A mixture of 2-amino-4-phenyl thiazole (0.02) and vanillin (0.02 mol) in ethenol (60 ml) and piperidine (3-4 drops) was refluxed on water bath for 4 hours. The reaction mixture was cooled and the solid separated was filtered and recrystallised from ethanol,



2-Amino-5-Chloro-4-(P0Subst. unsubst) Phenyl thiazole

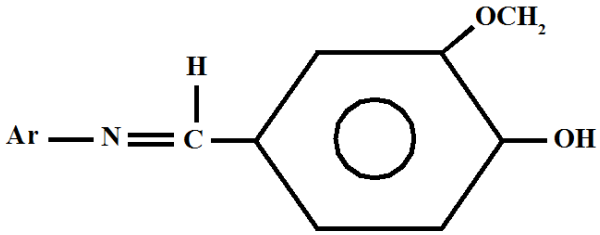
m.p. 150, yield 55% (Found N, 8.10, S 9.25; C17H13N2SO2Cl requires (N, 8.12; S, 9.28%) IR (KBr) 760-765 cm-1 (due to >C=O), 740-745 cm-1 (due to C-Cl), 6.90 cm-1 due to (C- S-C); 1640 and 1252 cm-1 (due to C = N and C-N); 1210-1220 cm-1, 1250 cm-1 (due to C-O-C), NMR; d3.82-3.95 (3H, s. OCH3), d4.52cm-1 (1H, d. CHCl).

Similarly various N-[5-Chloro-4-(P-subst/unsubst)-phenyl-2-thiazolyl]-2-amino-[3'-methoxy-4'-hydroxy]-benzylidine were prepared by using similar reaction procedurea and their analytical data are incorporate in the table (1).

N-[5-Chloro-4-Phenyl]-Thiazolyl-3"-chloro-4"-[4'-hydroxy-3'-methoxy phenyl]-2"-azetidinones-

Table (I)

Analytical data of N-[5-Chloro-4-(P-subst/un-subst) phenyl-2-thiazolyl]-2-imino-[3'-methoxy-4'-hydroxy-]benzylidine



Sl.No.	Nature of Ar.	Molecular Formula	Yield %	m.p. ^{0}C	% Analysis			
					N	S	N	S
Ia	2-Amino-5-chloro-4-phenyl thiazole	$C_{17}H_{13}N_2SO_2Cl$	45	150	8.09	9.25	8.12	9.28
Ib	2-Amino-5-chloro-4-(p-cholor)-phenyl thiazole	$C_{17}H_{13}N_2SO_2Cl2$	50	142	7.35	8.42	7.38	8.44
Ic	2-Amino-5-chloro-4-(p-Fluoro)-phenyl thiazole	$C_{17}H_{13}N_2SO_2ClF$	47	151	7.68	8.78	7.70	8.80
Id	2-Amino-5-chloro-4-(p-bromo)-phenyl thiazole	$C_{17}H_{13}N_2SO_2ClBr$	49	157	6.56	7.50	6.59	7.53
Ie	2-Amino-5-chloro-4-(p-nitro)-phenyl thiazole	$C_{17}H_{13}N_3SO_4Cl$	48	175	10.72	8.15	10.75	8.19
If	2-Amino-5-chloro-4-(p-hydroxy)- phenyl thiazole	$C_{18}H_{16}N_2O_3SCl$	46	220	7.42	8.50	7.45	8.52
Ig	2-Amino-5-chloro-4-(p-methyl)- phenyl thiazole	$C_{18}H_{16}N_2SO_2Cl$	49	209	7.75	8.85	7.78	8.90
Ih	2-Amino-5-chloro-4-(p-methox)- phenyl thiazole	$C_{18}H_{15}N_2O_2SCl$	51	185	7.40	8.50	7.45	8.52
II	2-Amino-5-chloro-4-(p-ethoxy)- phenyl thiazole	$C_{19}H_{18}N_2SO_3Cl$	50	235	7.15	8.19	7.18	8.21

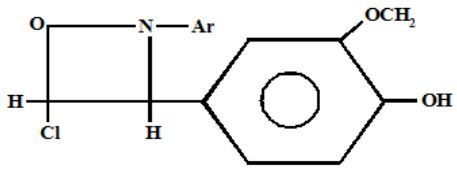
N-[5-Chloro-4-Phenyl]-Thiazolyl-3"-chloro-4"-[4'-hydroxy-3'-methoxy phenyl]-2"-azetidinones :

To a mixture of compound 1 (0.001 mol) and triethylamine (0.002 mol) dissolved in dioxane (25 ml), chloroacetyl chloride (0.0011 mol) was added dropwise at 100. The reaction mixture was stirred for 6 hours, then half of the solvent was removed by distillation and cooled, separated solid was recrystallised from chloroform to give m.p. 150 yield 45% (Found N 6.60 S 7.55 C19H14N2SO3Cl2 requires N 6.65; S 7.55%) ir (KBr) 1760-1765 cm-1 (due to >C = O, 740-745 cm-1 (due to C-Cl), 6.90 cm-1 due to (C- S-C); 1690 and 1252 cm-1 (due to C-O-C), NMR; d3.82-3.95 (3H, S. OCH3), d4.52cm-1 (1H, d. CHCl); d7.3-8.25 (8H, m. ArH) d4.1-4.6 cm-1 (1H, d-CH).

Similarly various N-[5-chloro-4-(P-subst/unsubst)-p-phenyl]-thiazolyl]-3"-chloro-4"-[3'-hydroxy-3'-methoxy phenyl]-2}-azetidinones were prepared by using similar reaction procedure and their analysis data are incorporated in the Table - II respectively.

Table (II)

Analytical Data of N-[5-Chloro-4-)p-subst/un-subst) phenyl]-thiazolyl-3"-chloro-4"-[4'-hydroxy-3'-methoxy-phenyl]-2]- azetidinones



Sl.No.	Nature of Ar.	Molecular Formula	Yield %	m.p. ^{0}C	% Analysis			
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Ic	2-Amino-5-chloro-4-(p-Fluoro)-phenyl thiazole	$C_{17}H_{13}N_2SO_2ClF$	47	151	7.68	8.78	7.70	8.80
Id	2-Amino-5-chloro-4-(p-bromo)-phenyl thiazole	$C_{17}H_{13}N_2SO_2ClBr$	49	157	6.56	7.50	6.59	7.53
Ie	2-Amino-5-chloro-4-(p-nitro)-phenyl thiazole	$C_{17}H_{13}N_3SO_4Cl$	48	175	10.72	8.15	10.75	8.19
If	2-Amino-5-chloro-4-(p-hydroxy)- phenyl thiazole	$C_{18}H_{16}N_2O_3SCl$	46	220	7.42	8.50	7.45	8.52
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II	2-Amino-5-chloro-4-(p-ethoxy)- phenyl thiazole	$C_{19}H_{18}N_2SO_3Cl$	50	235	7.15	8.19	7.18	8.21

ANTIFUNGAL ACTIVITY

In the present section the discussion have been made regarding the method used for determining the antifungal activity and the pharmacological screening, results of various compounds synthesised are given in the present investigation. A new azetidinones were evaluated for fungicidal activity by food and poison the following fungus are taken Alternaria alternata, curuvlaria lunata. Fusarium solani effect of newly synthesised antifungal compouds against these fungus at optimum tempr. of 28 ± 10C (After 7 days incubation) was observed.

Potato dextrose agar medium (20 cm3) prepared from potato 145g; dextrose 4.0g and agar 1.0 g in 170 cm3 distilled water) was poured into the sterilled petriplates and allowed to solidify. The plates were inoculated with spore suspenstion of either Alternaria alternata or curvularia lunata or Fusarium solani sp. (106 sporls cm3 of medium). By using a aterilised cork borer (9 cm-1 dia), wells were dug in the centre of the culture plates. The test solutions in DMSO were added (0.5 m3) to these Wells and the plates were incubated at 370 for 7 days. After seven days, the inhibition zone appearing around the wells in each plate was measured. To avoid the activity of the solvent, a solvent only treated plate was maintained. An untreated control was also maintained in order to calculate the percentage inhibition. Bavistin was used as a standard to compare the antifungal activity of the compounds lla-i.

Table - III

Antifungal Activity Data Inhibition (%)

Compound	Alternaria Alternata	Curvularia Iunata	Fusarium Solani
IIa	90.90	94.73	94.21
IIb	92.55	96.87	91.94
IIc	93.25	94.06	96.21
IId	94.24	94.24	90.58
IIe	94.73	93.09	91.42
IIf	90.39	91.94	92.71
IIg	89.05	90.78	90.38
IIh	93.20	91.21	90.28
IIi	95.39	94.40	90.29
BAVISTIN	99.65	98.02	99.58

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Cite this Article-

Roopali Tandon[1] & S.C. Mehra[2] "Synthesis of Some New Schiff bases with Possible Fungicidal Activity", Procedure International Journal of Science and Technology (PIJST), ISSN: 2584-2617 (Online), Volume:1, Issue:8, August 2024.

Journal URL- https://www.pijst.com/

DOI- https://doi.org/10.62796/pijst.2024v1i8003

Published Date- 05/08/2024