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SYNTHESIS AND CHARACTERIZATION OF (2E)-1-(PIPERAZIN-1-YL)-3-SUBSTITUTED PHENYLPROP-2-ONE CINNAMAMIDES

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ABSTRACT

Cinnamamides and their derivatives have a great era of its applications in medicinal as well as pharmaceutical fields. Several cinnamamides were isolated from plants and many of them are prepared in laboratory by different routes. In the present study different cinnamamides were synthesized by convenient witting reaction pathway from Wittig reagent with piperazine heterocyclic moiety and different aromatic aldehydes. All the synthesized compounds were characterized by using IR, ^{1}H-NMR, ^{13}C-NMR and Mass spectral analysis.

Keywords: *Piperazine, aromatic aldehydes, Cinnamamides.*

INTRODUCTION

Several cinnamamides and its derivatives were reported to shows variety of applications in different fields, such as medicinal, pharmaceuticals[1], agricultural and many other fields. Cinnamamides possess broad spectrum of physiological function and biological activities[2] and reported as Sedatives, nervous central system depressant[3], anticonvulsant, muscle relaxant, antiallergic, antioxidant[4], Local anesthetic[5], Antimycobacterial[6], Cytotoxicity[7] and Antioxidant[8]. The *N*-Feruloyl piperazine derivatives showed cytotoxic activity towards cancer cells and they have significant DNA binding activity[9]. In agrochemical field, their avian repellent[10], Antifungicidal, insecticidal and herbicidal activities[11]. This literature survey encourages the author to undertake the present research work and the Wittig reaction is an important method for the synthesis of cimamamides. In the present study, the attempts were made to synthesize the series of (2E)-1-(peperazin-1-yl)-3-substituted phenylprop-2-en-1-one Cinnamamides from Wittig reagent with piperazine moiety**.** Synthesized compounds were characterized by elemental analysis and spectral studies.

MATERIAL AND METHOD

Synthesis of Wittig reagent containing piperazine moiety-

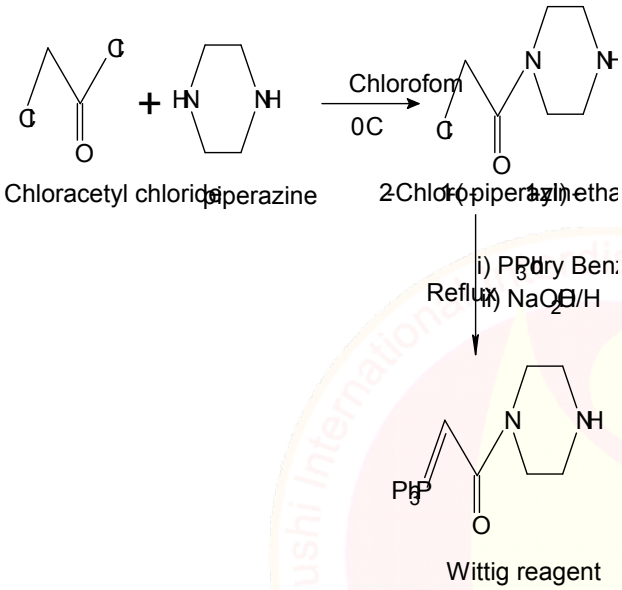
Piperazine chloracetamide were synthesized by using equimolar solution of chloro-acetylchloride and piperazine in chloroform at 0^{o}C with continuous stirring in fuming chamber. When this reaction mixture gives the salt by adding its solution in benzene to the stirred solution of triphenylphosphine and reaction mixture was refluxed for 4-6 hrs. The solid products obtained were filtered and air dried. Thus for Purification obtained salt was dissolved in 100 ml water then 90 ml of dry benzene, add 1-2 drops of phenolphthalein indicator and add NaOH solution in it till pink colour persist this was indicates that the neutralization of present acid from reagent. Then benzene layer was separated and washed with water and concentrated to one third volume. Finally the product scratched with n-Hexane to obtain solid Wittig reagent.

Synthesis of (2E)-1-(peperazin-1-yl)-3-Substituted phenylprop-2-en-1-one cinnamamides –

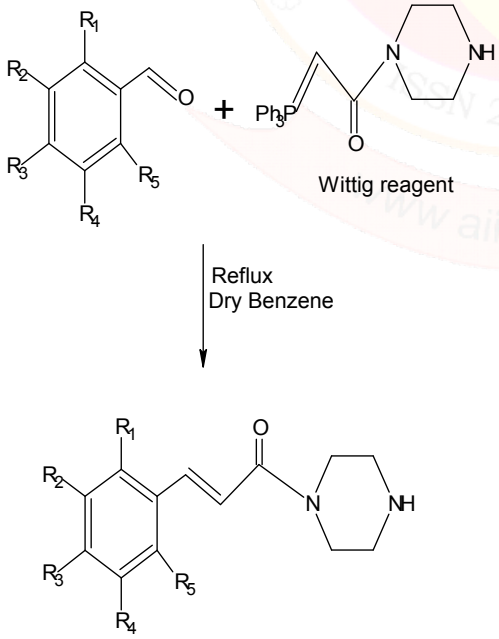
Equimolar solution of Wittig reagent and different aromatic aldehydes were taken in dry benzene and refluxed for 4 to 6 hrs. The progress of reaction was monitored by thin layer chromatography. Melting points were taken by open capillary method. The elemental analysis was calculated for

carbon, hydrogen, nitrogen and chlorine. All Synthesized compounds were purified by coloum chromatography. Obtained compounds were characterized by elemental analysis and spectral studies. All chemicals used were of analytical grade.

Scheme-1



Scheme-2



(2E)-1-(piperazin-1-yl)-3-subtituted phenylprop-2-en-1-one cinnamamides

Spectral Data Studies:-

Ia=(2E)-1-(peperazin-1-yl)-3- phenylprop-2-en-1-one Cinnamamides

^{1}H NMR (300 MHz, CDCl3) δ: 3.62-3.84 m 8H, 6.68 d (*J*= 15.2 Hz) 1H, 6.92d (*J*= 15.2 Hz) 1H, 7.36-7.56m 6H. **^{13}C NMR** (75 MHz, CDCl3) δ: 175, 136, 139, 130, 129, 128, 126, 122, 52, 48; **IR** (KBr, cm-1) :-3040, 1690, 1610; **MS** (ESI): 216.13(M^+). **Ib**=(E)-3-(*p*-Methoxyphenyl)-1-(1-piperazinyl)-2-propen-1-one Cinnamamide

^{1}H NMR-(δ):-3.81(s), (3H); 6.9d, (2H); 7.4d, (2H); 6.7(d), (1H), (CH=CHCO), J=15.8HZ; 7.6d, (1H), (CH=CHC$_6$H$_5$) J=15.8HZ; 7.2(d), (2H); 7.4(d), (2H). **^{13}C NMR** (δ ppm):166, 159, 136, 128, 114, 114, 55, 45. **IR (cm^{-1}):** 3070, 2925, 1475; **MS** (ESI): 246.14 (M^+).

Table-1:-Substituted aromatic aldehydes used in the synthesis of Cinnamamides

Sr. No	Entries	R1	R2	R3	R4	R5
1	**Ia**	H	H	H	H	H
2	**Ib**	H	H	OMe	H	H
3	**Ic**	H	OMe	OMe	H	H
4	**Id**	H	OMe	OMe	OMe	H
5	**Ie**	H	-O-CH$_2$-O-		H	H
6	**If**	NO_2	H	H	H	H
7	**Ig**	H	H	Cl	H	H
8	**Ih**	H	H	NO_2	H	H
9	**Ii**	H	H	$N(Me)_2$	H	H
10	**Ij**	H	H	OH	H	H

Table-2:- Characteristics data for synthesized Cinnamamides

Sr. No.	Entries	Molecular Formula	Molecular weight	Yield %	M.P. ^{0}C
1	**Ia**	$C_{13}H_{16}N_2O$	216	72	90
2	**Ib**	$C_{14}H_{18}N_2O_2$	246	84	136
3	**Ic**	$C_{15}H_{20}N_2O_3$	276	82	181
4	**Id**	$C_{16}H_{22}N_2O_4$	306	74	229
5	**Ie**	$C_{14}H_{16}N_2O_3$	260	70	201
6	**If**	$C_{13}H_{15}N_3O_3$	261	68	248
7	**Ig**	C_{13}H15ClN_2O	251	62	132
8	**Ih**	$C_{13}H_{15}N_3O_3$	261	71	226
9	**Ii**	$C_{15}H_{21}N_3O$	259	74	158
10	**Ij**	$C_{13}H_{16}N_2O_2$	232	80	274

Table-3:- Elemental Analysis of synthesized Cinnamamides

Sr. No.	Entries	Mol. Formula	Analysis (%) Found (Calculated)				
			% C	% H	% O	% N	% Cl
1	Ia	$C_{13}H_{16}N_2O$	72.19 (72.22)	7.46 (7.40)	7.40 (7.40)	12.95 (12.96)	--
2	Ib	$C_{14}H_{18}N_2O_2$	68.27 (68.29)	7.37 (7.32)	12.99 (13.00)	11.37 (11.38)	--
3	Ic	$C_{15}H_{20}N_2O_3$	65.27 (65.22)	7.30 (7.25)	17.37 (17.39)	10.14 (10.14)	--
4	Id	$C_{16}H_{22}N_2O_4$	62.73 (62.75)	7.24 (7.19)	20.89 (20.92)	9.14 (9.15)	--
5	Ie	$C_{14}H_{16}N_2O_3$	64.60 (64.62)	6.20 (6.15)	18.44 (18.46)	10.76 (10.77)	--
6	If	$C_{13}H_{15}N_3O_3$	59.76 (59.77)	5.79 (5.75)	18.37 (18.39)	16.08 (16.09)	--
7	Ig	$C_{13}H15ClN_2O$	62.28 (62.28)	6.03 (5.99)	6.38 (6.39)	11.17 (11.18)	14.14 (14.17)
8	Ih	$C_{13}H_{15}N_3O_3$	59.76 (59.77)	5.79 (5.75)	18.37 (18.39)	16.08 (16.09)	--
9	Ii	$C_{15}H_{21}N_3O$	69.47 (69.50)	8.16 (8.11)	6.17 (6.18)	16.20 (16.22)	--
10	Ij	$C_{13}H_{16}N_2O_2$	67.22 (67.24)	6.94 (6.90)	13.78 (13.79)	12.06 (12.07)	--

RESULT AND DISCUSSION

All synthesized novel cinnamamides compounds contained heterocyclic moiety in the form of Piperazine. The Wittig reaction is an important method for the synthesis of alkenes. By using this method novel cinnamamides containing heterocyclic moiety entitled (2E)-1-(Piperazin-1-yl)-3-substituted phenylprop-2-en-1-one Cinnamamides are synthesized from different aromatic aldehydes and Wittig reagents having good yields. The yields of synthesized compounds were ranging from 62 to 84%. All synthesized compounds were characterized on the basis of melting point, elemental analysis, IR spectra, ^{1}HNMR, ^{13}CNMR and mass spectral analysis.

CONCLUSION

The objective of the present study was to synthesize the novel cinnamamides containing heterocyclic moiety piperazine by using Wittig reagent and different aromatic aldehydes in dry benzene viz. Wittig reaction. The results of synthesized compounds were ranging from 62 to 84%. On the basis of melting point, elemental analysis, IR spectra, ^{1}HNMR, ^{13}CNMR and mass spectral analysis the characterization and yield of synthesized compounds, it was proved that given method is very good for synthesis of heterocyclic Cinnamamides.

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