Available online www.jocpr.com

Journal of Chemical and Pharmaceutical Research, 2012, 4(5):2681-2683

Research Article

ISSN : 0975-7384
CODEN(USA) : JCPRC5

Design and Spectral Studies of Novel Schiff Base derived from Pyrazole

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ABSTRACT

5-chloro-1,3-dimethyl-1h-pyrazole-4-carbaldehyde condensed with various aromatic amine. Finally the product were characterized by conventional and instrumental methods. Their structures were determined.

Keywords: Schiff base derivatives, Pyrazole, Spectral Studies.

INTRODUCTION

Azomethines are generally known as Schiff bases to honour Hugo Schiff, who synthesized such compounds. These are the compounds containing characteristic -C=N- group. Several methods have been reported for the preparation of azomethines. Selvam *et.al* [1] have prepared sulfonamide and its derivatives as anti-HIV agents. More *et. al* [2] have marked the biological activity of Schiff bases synthesized from aminothiazoles. Ernst Bayer [3] has reported some metallocomplex Schiff bases derived from *o*-amino phenol. Schiff bases can be synthesized from an aromatic amine and a carbonyl compound by nucleophilic addition forming a hemiaminal, followed by a dehydration to generate an imine [4]. They are well known intermediates for the preparation of azetidinones, thiazolidinones, oxadiazolines and many other derivatives. Azomethines exhibit a wide range of pharmacological activities like antimicrobial [5], antiparasitic [6], anti-inflammatory [7], anticancer [8] *etc*. The chemistry of pyrazole and its derivatives is particularly interesting because of their potential application in medicinal chemistry as analgesic, anti-inflammatory, antipyretic, antiparasitic, antimalarial, antifungal, antimicrobial and as enzyme inhibitory agents [9].

EXPERIMENTAL SECTION

The reagent grade chemicals were obtained from commercial sources and purified by either distillation or recrystallization before use. Purity of synthesized compounds has been checked by thin layer chromatography. Melting points were determined by open capillary method and are uncorrected. IR spectra are recorded on FT-IR Bruker with KBr disc. ^{1}H NMR spectra are recorded in DMSO-d6 on a Bruker DRX-400 MHz using TMS as internal standard. The chemical shift are reported as parts per million(ppm) and mass spectra were determined on Jeol-SX-102(FAB) spectrometer.

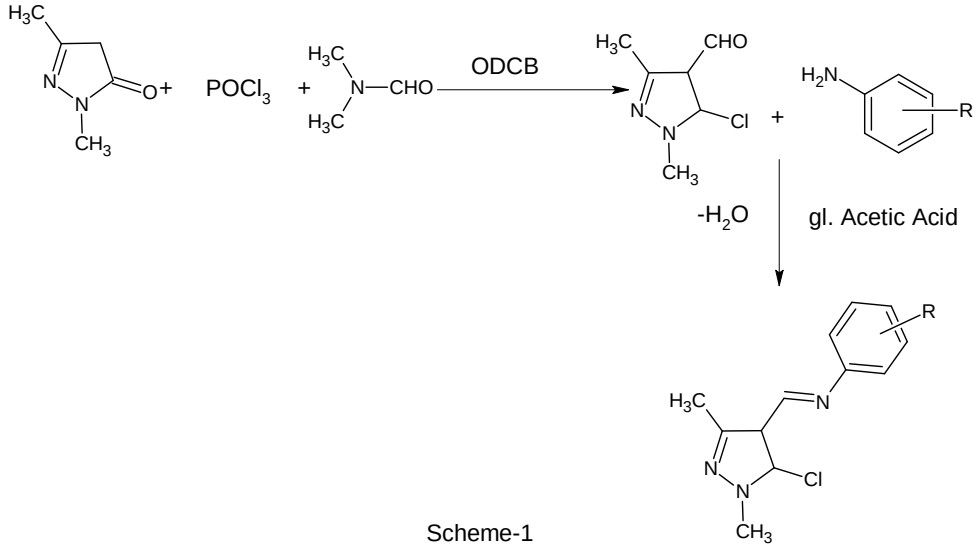
Synthetic Procedures

Preperation of 5-chloro-1,3-dimethyl-1h-pyrazole-4-carbaldehyde

Charge ODCB, DMF and DMPO in reactor, stir at 30°C for 20 minutes to get suspension. Cool the reaction mass at 5°C to 8°C. Add slowly $POCl_3$ (Duration: 5hrs.). After complete addition heat the reaction mixture slowly at 110°C and maintain the temperature upto 2hrs. Cool the reaction mass at 35°C. Prepared 8% NaHCO3 solution in reactor. Cool the sodium bicarbonate solution at 10°C. Add drop-wise $POCl_3$ mass into 8% NaHCO3 solution. Add MDC into the above reaction mass at RT and stirred for 30 minutes. Add 10% NaCl and stirred for 30 minutes and allow to settle for 30 minutes, separate the bottom organic layer. Add another MDC into aqueous phase and stir for 30 minutes, allow to settle 30 minutes, separate the bottom organic layer. Drum out the aqueous phase and note down the quantity. Organic layer was then concentrated via distillation in reactor. Drum out the organic layer.

Preparation of *N*-[(*E*)-(5-chloro-1,3-dimethyl-1*H*-pyrazol-4-yl)methylidene]-4-methylaniline

To a mixture of 5-chloro-1,3-dimethyl-1*H*-pyrazole-4-carbaldehyde (0.1 mol.) and substituted aromatic amine (0.1 mol.) in ethanol, catalytic amount of glacial acetic acid added then the resultant mixture was refluxed for (5-6 hours), progress of the reaction was monitored by TLC. After the completion of the reaction, the obtained product was poured into crushed ice stirred well; solid obtained was recrystallized from suitable solvent.



Scheme-1

Figure-1 Synthetic route of schiff-base derivatives

Table-1. Physical constants and elemental analysis of Schiff-base

Com. No.	Ar-	Molecular Formula	M.P °C	Yield %	% of C Found, (calcd.)	% of H Found, (calcd.)	% of N Found, (calcd.)
SP_{VIII}-1	4-Cl-C_6H_4	$C_{12}H_{11}Cl_2N_3$	156	77	53.77 (53.75)	4.15 (4.13)	15.68 (15.67)
SP_{VIII}-2	4-NO_2-C_6H_4	$C_{12}H_{11}ClN_4O_2$	141	79	51.73 (51.72)	3.99 (3.98)	20.11 (20.10)
SP_{VIII}-3	3,4-$(Cl)_2$-C_6H_3	$C_{12}H_{10}Cl_3N_3$	150	73	47.65 (47.63)	3.35 (3.33)	13.90 (13.89)
SP_{VIII}-4	3-NO_2-C_6H_4	$C_{12}H_{11}ClN_4O_2$	154	78	51.73 (51.72)	3.97 (3.98)	20.12 (20.10)
SP_{VIII}-5	2-OH-4-$NO_2C_6H_3$	$C_{12}H_{11}ClN_4O_3$	152	75	48.93 (48.91)	3.77 (3.76)	19.03 (19.01)
SP_{VIII}-6	2-OH-C_6H_4	$C_{12}H_{12}ClN_3O$	147	76	57.74 (57.72)	4.86 (4.84)	16.85 (16.83)
SP_{VIII}-7	2-Cl-C_6H_4	$C_{12}H_{11}Cl_2N_3$	149	80	53.77 (53.75)	4.15 (4.13)	15.69 (15.67)
SP_{VIII}-8	4-CH_3-C_6H_4	$C_{13}H_{14}ClN_3$	146	72	63.04 (63.03)	5.72 (5.70)	16.95 (16.96)
SP_{VIII}-9	2,4,5-$(Cl)_3$-C_6H_2	$C_{12}H_9Cl_4N_3$	162	79	42.77 (42.76)	2.68 (2.69)	12.48 (12.47)
SP_{VIII}-10	2-OCH_3-C_6H_4	$C_{13}H_{14}ClN_3O$	142	83	59.22 (59.21)	5.36 (5.35)	15.94 (15.93)
SP_{VIII}-11	2,4-$(NO_2)_2C_6H_3$	$C_{12}H_{10}ClN_5O_4$	148	79	44.55 (44.53)	3.12 (3.11)	21.66 (21.64)
SP_{VIII}-12	2,4-$(Cl)_2$-2 NO_2-C_6H_2	$C_{12}H_9Cl_3N_4O_2$	150	85	41.48 (41.47)	2.62 (2.61)	16.13 (16.12)
SP_{VIII}-13	3-Cl-6-OH-C_6H_3	$C_{12}H_{11}Cl_2N_3O$	159	75	50.73 (50.72)	3.92 (3.90)	14.80 (14.79)
SP_{VIII}-14	3-Cl-4-F-C_6H_3	$C_{12}H_{10}Cl_2FN_3$	143	78	50.39 (50.37)	3.33 (3.32)	14.71 (14.69)

Spectra study of *N*-[(*E*)-(5-chloro-1,3-dimethyl-1*H*-pyrazol-4-yl)methylidene]-4-methylaniline

IR(KBr. cm^{-1}):1622 cm^{-1}(C=N), 3053 cm^{-1}(C-H, str), 640 cm^{-1} (C-Cl), 1492 cm^{-1} (C=N, Pyrazole), 1288 cm^{-1} (C-N, Str.), 1006 cm^{-1} (N-N, Str.) ^{1}H NMR(ppm) ($CDCl_3$):6.62(s, 1H, N=CH), 6.64-8.03(m, 4H), 3.54(s, 3H, -CH_3), 2.56(s, 3H, -CH_3), MS:269[M+1].

RESULTS AND DISCUSSION

Various Schiff's base derivatives SP_{VIII} 1-1**4** were prepared using 5-chloro-1,3-dimethyl-1h-pyrazole-4-carbaldehyde with aromatic amine in presence catalytic amount of glacial acetic acid gave *N*-[(*E*)-(5-chloro-1,3-dimethyl-1*H*-pyrazol-4-yl)methylidene]-4-methylaniline. All the compounds synthesized were adequately characterized by their elemental analyses and spectral IR, ^{1}H-NMR and Mass Spectra.

CONCLUSION

As outline in Scheme-1, an important novel Schiff base *N*-[(*E*)-(5-chloro-1,3-dimethyl-1*H*-pyrazol-4-yl) methylidene]-4-methylaniline has been synthesized. All the structure of the above compounds were in good agreement with Spectral and Analytical data.

Acknowledgments
We are grateful the SAIF, Punjab University for recording the ^{1}H NMR, Oxygen Health care Research Pvt. Ltd., Ahmedabad for recording Mass Spectra and M.N. Science college, Patan for recording IR Spectra.

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