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Synthesis and Spectral Studies of some Novel Schiff Base derived with Pyrimidines

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ABSTRACT

4,6-dimethoxypyrimidin-2-amine condensed with various aromatic aldehyde. Finally the product were characterized by conventional and instrumental methods. Their structures were determined.

Keywords: Schiff base derivatives, Pyrimidines, Spectral study.

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*. Pyrimidine and their derivatives posseses several interesting biological activity such as antimicrobial [9-11], antitumor, antifungal activities. Many pyrimidine derivatives are used for thyroid drugs and leukemia.

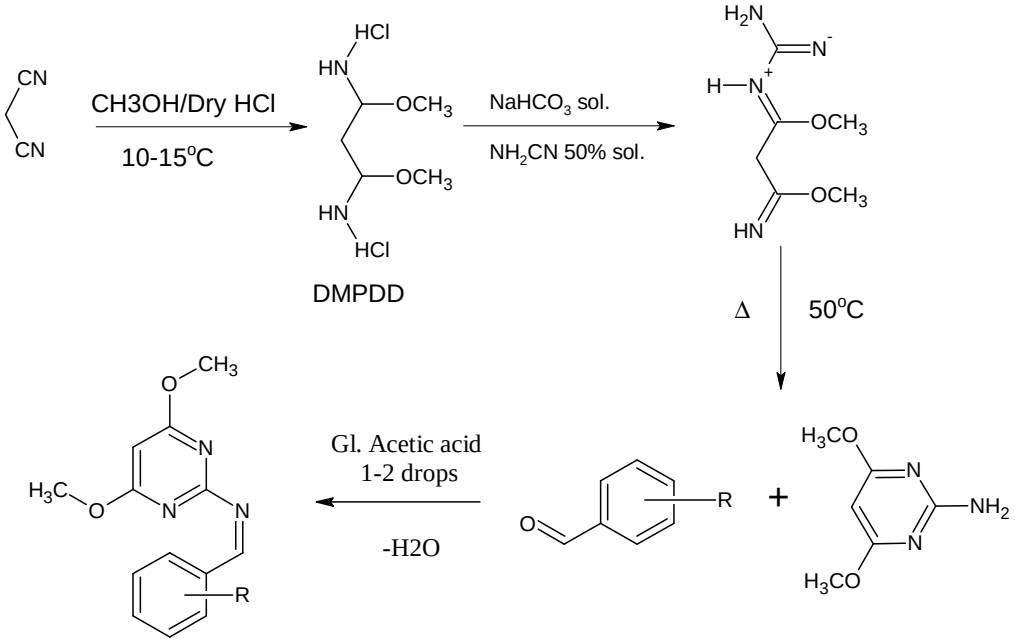
MATERIALS AND METHODS

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.

Preperation of 4,6-dimethoxy- pyrimidin-2-amine

Taken 2.0 liter 4 neck flask, temp pocket, mechanical stirrer, Charge Toluene, Malanonitrile, now RM cool to 10-15°C and dry HCl gas purging start, Continue pursing of dry HCl gas at 10-15°C temperature, till SM became absent, now filter the RM and W/C washed with toluene. Filter must be done in N_2 gas atmosphere because Di-imidat-Di-hydrochloride salt is highly hygroscopic. In another 2.0 lit. glass beaker taken NaHCO3 in water, then cool it 10-15°C temperature, now add di-hydrochloride salt as early as possible and maintain temperature 10-15°C, during addition of salt. Now add 50% cynamide solution at 10-15°C temperature, now slowly rise the temperature of RM till RT and stir for 4-5 hrs. If solid seems then filter it and suck dry well. In another 2.0 lit. 4 neck flask

taken Toluene and W/C, heat it till solid dissolve, now distilled out water azeotropically from the RM. Then distilled out approx 70-80% toluene and RM cool to 0°C temp., and filter it and W/C washed with chilled toluene, Suck dry well. Recrystallized from Methanol.



Scheme-1

Figure-1 Synthesis route of schiff-base derivatives

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

Comp. No.	-R	Molecular Formula	M.P °C	Yield %	% of C Found, (calcd.)	% of H Found, (calcd.)	% of N Found, (calcd.)
SP_I-1	-C_6H_5	$C_{13}H_{13}N_3O_2$	140	81	64.20 (64.19)	5.38 (5.39)	7.25 (7.27)
SP_I -2	4-OH-C_6H_4	$C_{13}H_{13}N_3O_3$	110	78	60.23 (60.22)	5.04 (5.05)	16.23 (16.21)
SP_I -3	4-Cl-C_6H_4	$C_{13}H_{12}ClN_3O_2$	170	70	56.24 (56.22)	4.35 (4.36)	15.15 (15.13)
SP_I -4	2-OH-C_6H_4	$C_{13}H_{13}N_3O_3$	290	74	60.20 (60.22)	5.03 (5.05)	16.22 (16.21)
SP_I -5	4-NO_2-C_6H_4	$C_{13}H_{12}N_4O_4$	120	77	54.18 (54.17)	(4.21) (4.20)	(19.45) (19.44)
SP_I -6	2,6-(CH_3)$_2$-C_6H_3	$C_{15}H_{17}N_3O_2$	145	79	66.41 (66.40)	6.33 (6.32)	15.48 (15.49)
SP_I -7	3,4,5-(OCH_3)$_3$-C_6H_2	$C_{16}H_{19}N_3O_5$	100	82	57.64 (57.65)	5.76 (5.75)	12.63 (12.61)
SP_I -8	2,4,5-(OCH_3)$_3$-C_6H_2	$C_{16}H_{19}N_3O_5$	145	77	57.66 (57.65)	5.78 (5.75)	12.60 (12.61)
SP_I -9	3,4-(OCH_3)$_2$-C_6H_3	$C_{15}H_{17}N_3O_4$	60	70	59.42 (59.40)	5.63 (5.65)	13.86 (13.85)
SP_I -10	2-Cl-C_6H_4	$C_{13}H_{12}N_3O_2$	260	81	56.23 (56.22)	4.37 (4.36)	12.78 (12.77)
SP_I -11	3- OCH_3-4- OH-C_6H_3	$C_{14}H_{15}N_3O_4$	100	73	58.14 (58.13)	5.24 (5.23)	14.52 (14.53)
SP_I -12	-CH-CH=CH- C_6H_5	$C_{15}H_{15}N_3O_2$	160	75	66.92 (66.90)	5.62 (5.61)	15.62 (15.60)
SP_I -13	-C_4H_2O	$C_{11}H_{11}N_3O_3$	140	76	56.66 (56.65)	4.76 (4.75)	18.04 (18.02)
SP_I -14	4-OCH_3-C_6H_4	$C_{14}H_{15}N_3O_3$	120	79	61.54 (61.53)	5.54 (5.53)	15.39 (15.38)
SP_I -15	2-NO_2-C_6H_4	$C_{13}H_{12}N_4O_4$	220	80	54.18 (54.17)	4.21 (4.20)	19.45 (19.44)

Preperation of 4,6-dimethoxy-*N*-[(*E*)-phenylmethylidene]pyrimidin-2-amine
To a mixture of 4,6-dimethoxy pyrimidine 2-amine (0.1 mol.) and substituted aromatic aldehyde (Benzaldehyde, 0.1 mol.) in methanol, 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. Their physical constant data are given in Table-1 and synthetic scheme in Figure-1.

Spectra study of 4,6-dimethoxy-*N*-[(*E*)-phenylmethylidene]pyrimidin-2-amine
IR(KBr. cm^{-1}):1623 cm^{-1}(C=N), 3060 cm^{-1}(C-H, str), 1247 cm^{-1} (C-O-C,str), 1430 cm^{-1} (C=N, Ar), ^{1}H NMR(ppm) ($CDCl_3$):8.32(s, 1H, N=CH), 6.81-7.82(m, 6H), 2.65(s, 6H), MS:243[M.].

RESULTS AND DISCUSSION

Various Schiff's base derivatives SP_I 1-15 were prepared using 4,6-dimethoxy-pyrimidine-2-amine with aromatic aldehyde(benzaldehyde) in presence catalytic amount of glacial acetic acid gave 4,6-dimethoxy-*N*-[(*E*)-phenylmethylidene]pyrimidin-2-amine. 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 4,6-dimethoxy-*N*-[(*E*)-phenylmethylidene]pyrimidin-2-amine has been synthesized. All the structure of the above compounds were in good agreement with Spectral and Analytical data.

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