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Design and spectral analysis of novel Schiff base derived with acetophenone derivatives

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ABSTRACT

6-Amino Indazole condensed with various aromatic acetophenone. Finally the product were characterized by conventional and instrumental methods. Their structures were determined.

Keywords: Schiff base derivatives, Spectral Analysis, Acetophenone derivatives.

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*.

Indazole and their derivatives display interesting biological properties and powerful pharmacological activities, such as anti-cancer, and anti-platelet activities, plus serotonin 5-HT3 receptor antagonist [9].

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.

Synthetic Procedures

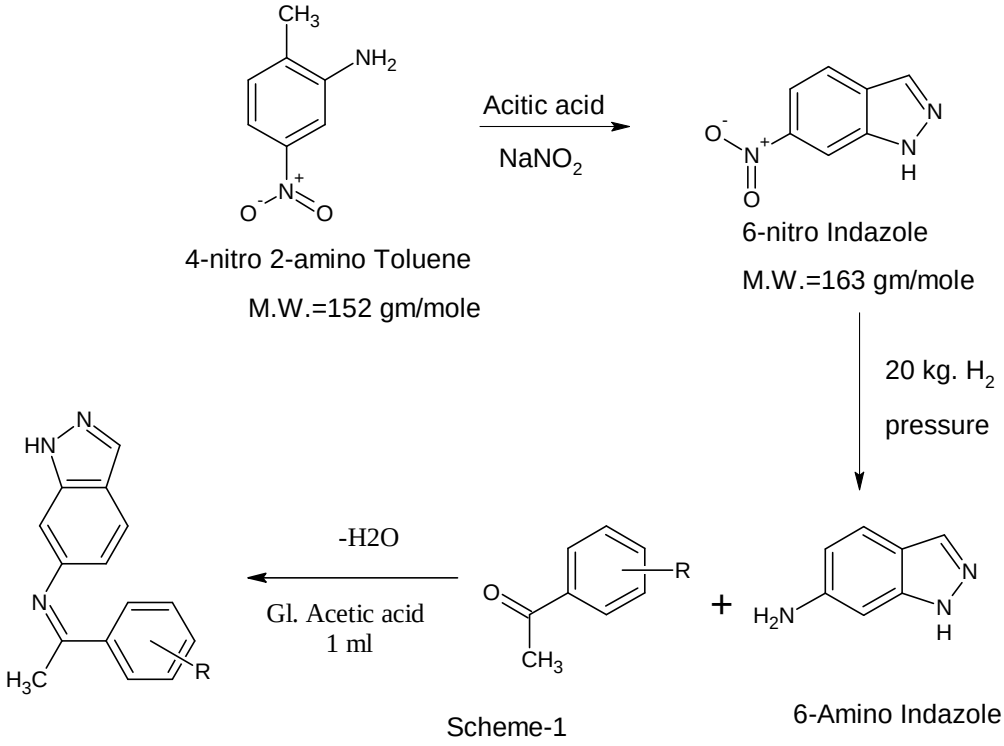
Preparation of 6-amino Indazole

Mix S.M. (4-Nitro 2-Amino tolvin) and acetic acid in 3-neck R.B.F. and stir for 30 min, Now add $NaNO_2$ and H_2O. Mixture in R.B.F. during 30 min. Now, temperature raise from 30°C to 55°C, then maintain temperature 70°C, Progress of the reaction was monitored by T.L.C. After the completion of the reaction, the obtained product was poured into chilled water stirred well, solid obtained was recrystallized from suitable solvent.

Mix 6 Nitro Indazole, Methanol and Raney Ni, in autoclave then apply 20.0 Kg. H_2 and heat it to 75^0C , continues H_2 pass, Progress of the reaction was monitored by T.L.C. After the completion of the reaction, the obtained product was poured into chilled water stirred well, solid obtained was recrystallized from suitable solvent.

Preparation of *N*-[(1*Z*)-1-(4-aminophenyl)ethylidene]-1*H*-indazol-6-amine

To a mixture of 6-amino Indazole (0.1 mol.) and substituted aromatic acetophenone (4-amino acetophenone, 0.1 mol.) in ethanol, 1ml. 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.



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_{VII}-1	4-NH_2-C_6H_4	$C_{15}H_{14}N_4$	80	78	71.99 (71.98)	5.65 (5.64)	22.39 (22.38)
SP_{VII} -2	3-OCH_3-4-OH-C_6H_3	$C_{16}H_{15}N_3O_2$	65	70	68.30 (68.31)	5.39 (5.37)	14.95 (14.94)
SP_{VII} -3	3-F-C_6H_4	$C_{15}H_{12}F\ N_3$	68	74	71.14 (71.13)	4.79 (4.78)	16.57 (16.59)
SP_{VII} -4	3-OH-C_6H_4	$C_{15}H_{13}N_3O$	70	77	71.72 (71.70)	5.19 (5.21)	16.71 (16.72)
SP_{VII} -5	2,4-$(OH)_2$-C_6H_3	$C_{15}H_{13}N_3O_2$	54	82	67.41 (67.40)	4.92 (4.90)	15.74 (15.72)
SP_{VII} -6	2,4-$(Cl)_2$-5-F-C_6H_2	$C_{15}H_{10}Cl_2FN_3$	55	70	55.93 (55.92)	3.15 (3.13)	13.05 (13.04)
SP_{VII} -7	2,6-$(Cl)_2$-3-F-C_6H_2	$C_{15}H_{10}Cl_2FN_3$	58	73	55.93 (55.92)	3.15 (3.13)	13.05 (13.04)
SP_{VII} -8	3-Cl-4-F-C_6H_3	$C_{15}H_{11}ClFN_3$	60	82	62.63 (62.62)	3.86 (3.85)	14.62 (14.60)
SP_{VII} -9	3-F-C_6H_4	$C_{15}H_{12}FN_3$	62	79	71.14 (71.13)	4.79 (4.78)	16.58 (16.59)
SP_{VII} -10	2-Br-4-F-C_6H_3	$C_{15}H_{11}BrFN_3$	68	80	54.25 (54.24)	3.35 (3.34)	12.67 (12.65)

Spectra study of *N*-[(1*Z*)-1-(4-aminophenyl)ethylidene]-1*H*-indazol-6-amine
IR(KBr. cm^{-1}):1589 cm^{-1}(C=N), 1280 cm^{-1} (C-N, Primary –NH_2), 3354 cm^{-1} & 3333 cm^{-1}(N-H, Str.), 1653 cm^{-1} (N-H, bending), 3080 cm^{-1}(C-H, str), 1564 cm^{-1} (C=N, Indazole), ^{1}H NMR(ppm) ($CDCl_3$):7.67-7.69(s, 2H, NH_2), 6.53-7.38(m, 7H), 2.40-2.48(s, 3H, -CH_3), MS:251[M+1].

RESULTS AND DISCUSSION

Various Schiff's base derivatives SP_{VI} 1-15 were prepared using 6-amino Indazole with aromatic acetophenone (4-amino acetophenone) in presence of 1 ml. glacial acetic acid gave *N*-[(1*Z*)-1-(4-aminophenyl)ethylidene]-1*H*-indazol-6-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 *N*-[(1*Z*)-1-(4-aminophenyl)ethylidene]-1*H*-indazol-6-amine has been synthesized. All the structure of the above compounds were in good agreement with Spectral and Analytical data.

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