



Synthesis and Spectral Characterization of Some New Novel Schiff bases Derived From Hydroxy Propiophenone

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ABSTRACT

New Schiff bases derived by the condensation of methyl, chloro and bromo substituted 2' hydroxy propiophenones with aliphatic and aromatic amines have been synthesized. The Schiff bases are yellow solids with sharp melting points. The spectral characterization of these newly synthesized Schiff base ligands has been done. These Schiff bases were characterized by IR spectroscopy, Nuclear magnetic resonance spectroscopy, UV-spectroscopy and Mass spectroscopy. IR-spectra confirmed the presence of the phenolic OH group and azomethine group.

Keywords: Substituted hydroxy propiophenone, Aromatic amines, Aliphatic amines, NMR, Mass, Schiff base ligands.

INTRODUCTION

Schiff bases constitute the important class of organic compounds containing azomethine group (-CH=N-) which is generally obtained by the condensation of aromatic aldehydes or ketones with amines. Nowadays research dealing with the coordination complexes of Schiff bases with various metal ions has expanded enormously. Schiff bases act as a good chelating agent¹ and exhibit extraordinary properties with metal complexes. Some heterocyclic Schiff base ligands form metal chelates² and play a very important role in medicinal chemistry. Schiff bases due to their wide range of applications becoming more and more interesting for the researchers. The Schiff bases are very much important in bioinorganic chemistry, biomedical chemistry,³⁻⁴ pharmaceutical industries,

catalysis⁵⁻⁹, polymer chemistry, supramolecular chemistry, catalysis, material science. They have been found to possess antimicrobial, antifungal, antimalarial, antiviral activities¹⁰⁻¹⁵. Recently Schiff bases having halogen groups and their metal complexes have been found to possess special interest due to their pharmacological properties. These metal complexes of Schiff base shows various biological activities¹⁶⁻¹⁷. These are also used as anticancer agents. Generally, ketones show less reactivity than aldehydes. A large number of Schiff bases were synthesized from hydroxy aldehydes but lesser work has been done on hydroxy propiophenone. Schiff bases act as an effective chelating agent when it has an OH group in the vicinity of the azomethine (>C=N-) group and forms metal complexes that possess outstanding biological properties.



Because of the biological importance of Schiff bases, we have synthesized some new Schiff bases from chloro hydroxy propiophenone, bromo hydroxy propiophenone, and methyl hydroxy propiophenone with benzylamine, p-toluidine, aniline and propylamine and these newly prepared ligands were characterized by spectral analysis.

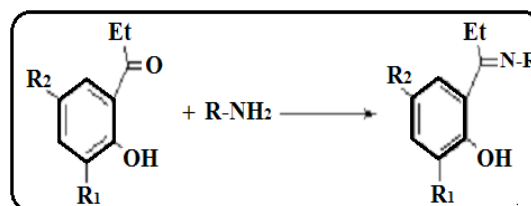
MATERIAL AND METHOD

Chemicals used were of analytical grade without further purification. Iodination of bromo and chloro 2' hydroxypropiophenone was done by using iodine and iodic acid¹⁸. Melting points were determined by an open capillary tube and were uncorrected. Thin layer chromatography was used to determine the progress of the reaction. ¹H NMR spectra of all the ligands were recorded in DMSO solvent using Bruker Avance Neo 500MHz spectrophotometer, FTIR spectra were recorded on Shimadzu spectrometer and the mass spectra was recorded on Q-TOFMicromass (ESI-MS). UV-spectra were recorded on a W-1800 series spectrometer.

General Procedure for the synthesis of Schiff bases

All the Schiff base ligands were synthesized by refluxing substituted hydroxy propiophenone

(0.01M) with different aliphatic and aromatic amines (0.01M) for 3-5 hours. The reaction was monitored after the regular interval by TLC. When the reaction was complete, cooled the resulting mixture at room temperature and then transferred in ice-cold water. The yellow product separated which was then recrystallized by hot ethanol.



Synthesis of Schiff bases

RESULT AND DISCUSSION

The analytical and physical data of Schiff base ligand are given in the following Table 1. All the ligands were yellow colored, solid and stable at room temperature. The UV-spectra of the ligands showed the strong absorption bands at 330-350 nm and 280-290 nm which were attributed to n-n* and π-π* transitions respectively. Strong IR bands were observed in the range 3000-3100 cm⁻¹ which is the characteristic of the phenolic OH group. The details of the spectroscopic data are illustrated below the table.

Table 1: Physical and Analytical Data of Schiff base Ligand

Ligand	R ₁	R ₂	R	Melting Point (°C)	Yield (%)	Elemental Analysis (%) calculated (found)		
						C	H	N
2a	H	Br	C ₆ H ₅ CH ₂	110	68	60.39(60.56)	5.10(5.04)	4.35(4.41)
2b	I	Br	C ₆ H ₅ -CH ₂	122	62	36.39(36.54)	3.72(3.80)	3.44(3.55)
3a	H	CH ₃	C ₆ H ₅	131	66	80.20(80.33)	7.16(7.11)	5.75(5.85)
3b	I	Br	C ₆ H ₅	130	55	41.89(41.95)	3.08(3.03)	3.36(3.26)
4a	H	CH ₃	C ₆ H ₄ -OCH ₃	85	54	80.50(80.63)	7.56(7.50)	5.43(5.53)
4b	I	Cl	C ₆ H ₄ -OCH ₃	125	55	48.14(48.06)	3.82(3.75)	3.55(3.5)
5a	H	Br	n-C ₃ H ₇	82	54	53.35(53.53)	5.77(5.94)	5.08(5.20)
5b	H	Cl	n-C ₃ H ₇	72	61	70.32(70.20)	5.91(5.85)	5.22(5.11)
6a	H	Cl	Iso-C ₃ H ₇	90	65	70.32(70.20)	5.91(5.85)	5.22(5.11)
6b	I	Cl	Iso-C ₃ H ₇	110	40	40.89(41.08)	4.23(4.27)	3.67(3.99)

2a) 2-(1-(benzyl imino) propyl)4-bromophenol
IR (KBr): 3028.37 (OH), 1604.85 (C=N), 1284.65 (C-O) cm⁻¹. ¹H NMR (DMSO): -δ 1.16(t, 3H), 2.97 (q, 2H), 4.88(s, 2H), 6.63-7.79(m, Ar-H), 16.86(s, 1H, OH) ppm Mass:- m/z-317M⁺.

2b) 2-(1-(benzyl imino) propyl)4-bromo-6-iodo phenol
IR (KBr): 3078.52(OH), 1612.56(C=N), 1284

.65(C-O) cm⁻¹. ¹H NMR (DMSO): -δ 1.07(t, 3H), 3.18v (q, 2H), 3.32(s, 2H), 6.63-7.79(m, Ar-H), 12.97 (s, 1H, OH) ppm Mass:-m/z-443 M⁺.

3a) 4-methyl-2-(1-phenylimino)propyl phenol
IR (KBr): 3055.38(OH), 1597.13(C=N), 1203.63(C-O) cm⁻¹. ¹H NMR (DMSO): -δ 1.16(t, 3H), 2.23(q, 2H), 6.63-7.79(m, Ar-H), 14.45(s, 1H, OH) ppm Mass:-m/z-239M⁺

3b)4-bromo-2-iodo-6-(1-phenylimino)propyl phenol

IR(KBr): 3045.38(OH), 1651.14(C=N), 1273.07(C-O) cm^{-1} . $^1\text{H NMR}$ (DMSO) :- δ 1.17(t, 3H), 2.50(q, 2H), 7.35-7.84(m, Ar-H), 17.78(s, 1H, OH)ppm
Mass:-m/z-429 M^+

4a) 2-(1-p -tosylimino) propyl)4-methyl phenol

IR(KBr): 3065.51(OH), 1643.42(C=N), 1273.07(C-O) cm^{-1} . $^1\text{H NMR}$ (DMSO) :- δ 1.21(t, 3H), 2.99(q, 2H), 2.35(s, 6H, Ar- CH_3), 7.08-7.30(m, Ar-H), 16.00(s, 1H, OH)ppm
Mass:-m/z-253 M^+

4b)2-(1-p-tosylimino) propyl)4-chloro-6-iodo phenol

IR(KBr) : 3063.09(OH), 1651.14(C=N), 1265.36(C-O) cm^{-1} . $^1\text{H NMR}$ (DMSO) :- δ 1.16(t, 3H), 2.02(q, 2H), 1.01(s, 3H), 6.67-7.79(m, Ar-H), 15.55(s, 1H, OH)ppm
Mass:-m/z-399 M^+

5a)4-bromo-2-(E)-1-(propyl imino) propyl phenol

IR(KBr): 3070.81(OH), 1643.42(C=N), 1273.07(C-O) cm^{-1} . $^1\text{H NMR}$ (DMSO) :- δ 1.01(t, 3H), 1.16(t, 3H), 2.01(m, 2H), 3.89(t, 2H), 6.45-8.19(m, Ar-H), 15.86(s, 1H, OH)ppm
Mass:-m/z-269 M^+

5b) 4-chloro-2-(E)-1-(propyl imino) propyl phenol

IR(KBr): 3068.52(OH), 1612.56(C=N), 1262.43(C-O) cm^{-1} . $^1\text{H NMR}$ (DMSO) :- δ 1.01(t, 3H), 1.11(sex, 2H), 2.83(t, 2H), 8.13-8.19(m, Ar-H), 17.10(s, 1H, OH)ppm
Mass:-m/z-225 M^+

6a) 4-chloro-2-(1-isopropyl imino) propyl phenol

IR(KBr): 3075.55(OH), 1602.16(C=N), 1284.65(C-O) cm^{-1} . $^1\text{H NMR}$ (DMSO) :- δ 1.03(t, 3H), 1.95(q, 2H), 3.48(m, 1H), 6.70-7.48(m, Ar-H), 15.45(s, 1H, OH)ppm
Mass:-m/z-225 M^+

6b) 4-chloro-2-iodo 6-(1-isopropyl imino) propyl phenol

IR(KBr): 3078.52(OH), 1612.56(C=N), 1284.65(C-O) cm^{-1} . $^1\text{H NMR}$ (DMSO) :- δ 1.16(t, 3H), 2.04(q, 2H), 3.87(m, 1H), 6.87-7.67(m, Ar-H), 16.06(s, 1H, OH)ppm
Mass:-m/z-350 M^+

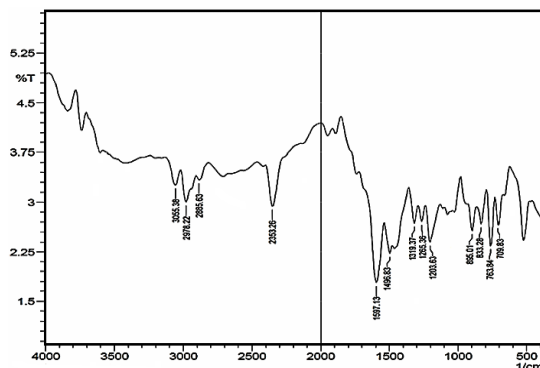


Fig. 1. IR spectra of 3a

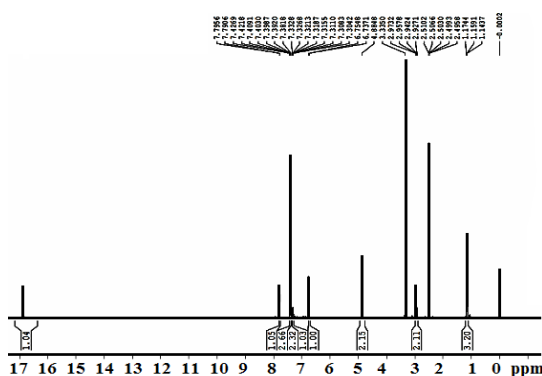
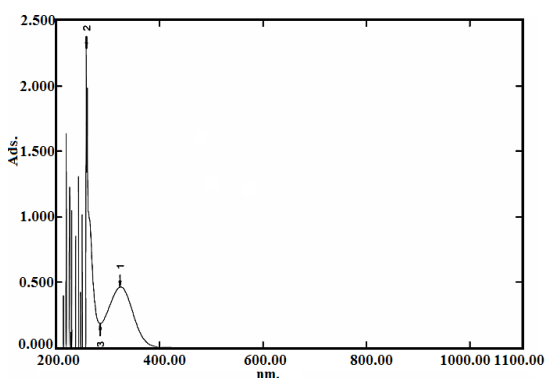
Fig. 2. $^1\text{H NMR}$ spectra of 2a

Fig. 3. UV spectra of 2b

CONCLUSION

New Schiff base ligands were synthesized by reacting substituted 2' hydroxy propiophenone with different aromatic and aliphatic amines. All the ligands showed a strong IR band at 3000-3200 cm^{-1} for phenolic -OH, a band at 1684-1595 cm^{-1} for C=N stretch. All the structures were confirmed by NMR and mass spectra. NMR studies showed the presence of OH peak at 12 to 17 ppm in all the ligands.

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Conflicts of Interest

The authors declare no conflict of interest.

REFERENCES

1. Krishnapriya K.R.; Kandaswamy M., *Polyhedron.*, **2005**, *24*,113.
2. Abd El-Halim H. F. Mohamed G. G.; Anwar M. N., *Appl Organometal Chem.*, **2017**, e3899.
3. Saito H.; Hoffmann A.S. Ogazoa H.I.J.; *Bioact. Compd. Polym.*, **2007**, *22*, 589.
4. In G.; Kim Y and Choi J., *Bull. Korean Chem. Soc.*, **2008**, *29*(5), 969.
5. Panja A.; Shaikh N.; Ali M.; Vojtisek P.; Banerjee P., *Polyhedron.*, **2003**, *22*, 1191.
6. Djebbar S. S.; Benali B.O.; Deloume J. P.; *Trans. Metal Chem.*, **1998**, *23*, 443.
7. Xishi Tai Xianhong Y.; Qiang C.; Minya T.; *Molecules.*, **2003**, *8*, 49.
8. Wang K.; Wedeking K.; Zuo W.; Zhang D.; Wen-Hua Sun, *J.Organomet Chem.*, **2008**, *693*(6), 1073.
9. Long Z.; Wu B.; Yang P.; Gang Li, Liu Y.; Xiao-Juan Y., *J. Organomet. Chem.*, **2009**, *694*(32), 3793.
10. Ommenya, F. K.; Nyawade, E. A.; Andala, D. M and Kinyua, J., *Journal of Chemistry*, **2020**, 1745236.
11. Spinu C.; Pleniceanu M.; Tigae C. *Turkish J Chem.*, **2008**, *32*(4), 487-493.
12. Zafar H.; Kareem A.; Sherwani A., *J Photochem Photobiol B Biol.*, **2015**, *142*, 8-19.
13. Hueso-Urena F.; Illan-Cabeza NA.; Moreno-Carretero MN.; Martinez-Martos JM.; Ramnirez-Exposito MJ. *J. Inorg. Biochem.*, **2003**, *94*(4), 326-334.
14. Abd El-halim H. F.; Omar M. M.; Mohamed G. G., *Spectrochim. Acta*, Part A, **2011**, *78*, 36-44.
15. Hameed A.; Al-Rashida M.; Uroos M.; Abid Ali S.; Khan K. M.; *Expert Opin. Ther. Pat.*, **2017**, *27*, 63-79.
16. Samina K.T.; Abhijit A.Y.; Ratnamala S.B. *J. Mol. Struct.*, **2018**, *1152*, 223-231.
17. Kursunlu A.N.; Guler E.; Sevgi F.; Ozkalp B., *J. Mol. Struct.*, **2013**, *1048*, 476-481.
18. Patil B.R.; Bhusare S.R., *Pawar Tetrahedron Letters*, **2005**, *46*(42), 7179-7181.