

## SHORT COMMUNICATION

### CONVENIENT AND MILD SYNTHESIS AND CHARACTERISATION OF SOME NEW SCHIFF BASES

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**ABSTRACT.** Some new Schiff bases were synthesized by the condensation between 2-hydroxy-1-naphthaldehyde and various diamines in 2:1 molar ratio. These pure products were prepared in methanol solution under mild reaction conditions to afford good to high yields. The structures of the products have been characterized by several techniques using elemental analysis (CHN), FT-IR, <sup>1</sup>H NMR, <sup>13</sup>C NMR and mass spectra.

**KEY WORDS:** Schiff base, 2-Hydroxy-1-naphthaldehyde, Diamine, Condensation, Synthesis

## INTRODUCTION

Schiff bases are reagents, which are becoming increasingly important in the pharmaceutical, dye and plastic industries as well as for liquid-crystal technology and mechanistic investigations of the drugs used in pharmacology, biochemistry, physiology, food chemistry and nanotechnology [1]. Schiff bases with donors (N, O, S, etc.) have structural similarities with natural biological systems and imports in elucidating the mechanism of transformation reaction in biological systems due to presence of imine (-N=CH-) group [2-10]. Schiff base complexes have been used as drugs and they possess a wide variety of antimicrobial activity against bacteria, fungi and certain type of tumors [11, 12]. Some drugs have increased activity when administered as metal chelates and inhibit the growth of tumors [13, 14].

Tetradentate Schiff bases are well known to coordinate with various metal ions forming stable compounds and some of these complexes are recognized as oxygen carriers [15]. These compounds also find many important catalytic applications in chemistry. They are used as the catalyst in reactions. These compounds also find many important catalytic applications, ranging from asymmetric epoxidation [16], Lewis acid assisted organic transformations [17], solid phase extraction of metal ions [18] to various types of polymerization [19-22] as well as their application for the preparation of ion selective electrodes [23-27]. Recently, Ayla Balaban Gunduzalp and co-worker have been prepared and characterized complexes of tetra dentate Schiff base ligands [28].

In continuation of our research on the synthesis of Schiff bases and their complexes of metal ions [29-31], we have studied the preparation of several new Schiff bases from the reaction of 2-hydroxy-1-naphthaldehyde with various diamines in methanol solution under mild conditions. The corresponding products were identified and characterized by spectroscopic and physical data.

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## EXPERIMENTAL

**Materials.** Chemical were purchased from the Merck Chemical Company in high purity. All the materials were of commercial reagent grade. The various amines and aldehydes and the solvents used were purified by standard procedures.

**Apparatus.** IR spectra were obtained as KBr pellets on a Perkin–Elmer 781 spectrophotometer and on an Impact 400 Nicolet FTIR spectrophotometer.  $^1\text{H}$  NMR spectra were recorded in  $\text{CDCl}_3$  on a (400 MHz) Spectrometer using TMS as an internal reference. Melting points were obtained with a Yanagimoto micro melting point apparatus are uncorrected. Mass spectra were recorded on Micro Mass UKLTD spectra. The purity determination of the substrates and reaction's monitoring were accomplished by TLC on silica-gel polygram SILG/UV 254 plates.

**General procedure for the synthesis of Schiff base ligands.** All the tetra dentate Schiff base ligands were prepared by condensation between diamines and 2-hydroxy-1-naphthaldehyde in methanol and purified by recrystallization from a methanol through the partial evaporation of the more volatile dichloromethane. Schiff base ligands prepared by dissolving 2-hydroxy-1-naphthaldehyde (0.5 g, 2.9 mmol) and diamine (0.15 g, 1.45 mmol) in methanol with stirring in one portion. The stirring was continued to completion of the reaction. The progress of the reaction was monitored by thin layer chromatography (TLC). After the completion of the reaction, a colored substance was obtained. The solid product was filtered and washed with cold MeOH. The crude product was purified by recrystallization from methanol and the pure Schiff base was obtained in high yield after leaving for the appropriate time. The Schiff bases were characterized by physical and spectroscopic data [15].

**2,2'-[1,2-Ethanediybis(nitrilomethylidene)]bis-(2-naphthol) (3a).** Yellow solid, m.p. = 330 °C; IR (KBr),  $\text{v}/\text{cm}^{-1}$ : 3200-3450 (stretch, OH, H-bonded), 1640 (stretch, C=N), 1490-1540 (C=C, Ar), 1207 (stretch, C-O);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  (ppm): 14.09 (s, 2O-H, intramolecular hydrogen bonding), 8.44 (s, 2CH imine), 7.75 (d, 2H, Ar), 7.31(d, 2H, Ar), 7.11 (d, 2H), 7.04 (t, 2H, Ar), 6.93 (t, 2H), 6.74 (d, 2H), 3.46 (s, 4H, 2CH<sub>2</sub>);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  (ppm): 159.24, 138.05, 136.46, 128.26, 127.76, 126.26, 125.88, 123.32, 118.14, 106.73, 76.45, 51.98, 31.06, 28.28; anal. calcd.: 77.81 (C), 5.99 (H), 7.56 (N); found: 77.83 (C), 5.67 (H), 7.37 (N).

**1,1'-[1,7-Heptanediybis(nitrilomethylidene)]bis-(2-naphthol) (3b).** Yellow solid, m.p. = 172-173 °C; IR (KBr),  $\text{v}/\text{cm}^{-1}$ : 3200-3500 (stretch, OH, H-bonded), 1632 (stretch, C=N), 1457-1542 (C=C, Ar), 1138, 1181 (stretch, C-O);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  (ppm): 14.35 (s, 2O-H, intramolecular hydrogen bonding), 8.5 (s, 2CH imine), 7.6 (d, 2H, Ar), 7.49(d, 2H, Ar), 7.4 (d, 2H), 7.24 (t, 2H, Ar), 7.03 (t, 2H), 6.77 (d, 2H), 3.4 (t, 4H, 2CH<sub>2</sub>) 1-2 (m, 10H, 5CH<sub>2</sub>);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  (ppm): 155.76, 138.35, 133.23, 128.26, 127.23, 125.44, 124.58, 121.13, 115.68, 104.49, 75.45, 51.81, 29.79, 27.08, 26.98; mass ( $m/z$ ): 441 ( $M^+ + 1$ , 3), 440 ( $M^+$ , 12), 281 (10), 198 (15), 143 (20), 152 (35), 184 (70), 170 (77), 115 (100).

**1,1'-[1,8-Octanediybis(nitrilomethylidene)]bis-(2-naphthol) (3c).** Yellow solid, m.p. = 180-183 °C; IR (KBr),  $\text{v}/\text{cm}^{-1}$ : 3250-3600 (stretch, OH, H-bonded), 3049 (stretch, C-H, Aryl), 2852, 2924 (stretch, C-H, alkyl), 1634 (stretch, C=N), 1457-1542 (C=C, Ar), 1181 (stretch, C-O);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  (ppm): 14.2 (s, 2O-H, intramolecular hydrogen bonding), 8.50 (s, 2CH imine), 1.20-1.60 (m, 4H, 2CH<sub>2</sub>), 3.50 (m, 12H, 6CH<sub>2</sub>), 7.66 (d, 2H, Ar), 7.5(d, 2H), 7.42 (d, 2H), 7.25 (t, 2H) 7.06 (m, 2H), 6.76 (d, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  (ppm): 157.58, 137.37, 133.88, 129.25, 127.95, 126.11, 125.32, 122.63, 117.69, 106.38, 76.73, 52.82, 30.58,

28.80, 26.63; mass (m/z): 453 ( $M^+ + 1$ , 0.1), 452 ( $M^+$ , 10), 295 (10), 198 (12), 143 (15), 166 (17), 184 (68), 170 (75), 115 (100).

*1,1'-1,2-Phenylenediylbis(nitrilomethylidene)]bis-(2-naphthol) (3d)*. Orange solid, m.p. = 162-163 °C; IR (KBr),  $\nu/\text{cm}^{-1}$ : 3373-3470 (stretch, OH, H-bonded), 3046 (stretch, C-H, aryl), 1612 (stretch, C=N), 1487-1559 (C=C, Ar), 1169 (stretch, C-O);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  (ppm): 15.45 (s, 2O-H, intramolecular hydrogen bonding), 9.39 (s, 2CH imine), 8.29 (d, 2H, J = 8) 7.73-7.61 (dd, J = 8, 4H, Ar), 7.34 (t, 2H, J = 8), 7.25 (d, 2H, Ar, J = 4), 7.15 (t, 2H, Ar, J = 4), 6.87 (t, 2H, Ar, J = 8), 6.63 (d, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  (ppm): 170.02, 153.44, 142.15, 137.56, 135.14, 132.26, 131.62, 129, 71, 128.20, 127.12, 124.29, 122.55, 119.24, 114.37; anal. calcd.: 80.36 (C), 5.30 (H), 6.69 (N); found: 80.31(C), 6.67 (H), 6.78 (N).

*1,1'-1,4-Phenylenediylbis(nitrilomethylidene)]bis-(2-naphthol) (3e)*. Orange solid, m.p. = 189-191 °C; IR (KBr),  $\nu/\text{cm}^{-1}$ : 3250-3500 (stretch, OH, H-bonded), 3200 (stretch, C-H, aryl), 1620 (stretch, C=N), 1512 (C=C, Ar), 1284 (stretch, C-O);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  (ppm): 15.09 (s, 2O-H, intramolecular hydrogen bonding), 9.40 (s, CH imine), 7.74 (d, 2H), 7.69, 7.54 (dd, 8H), 7.58 (t, 2H), 7.17 (t, 2H), 6.78 (d, 2H), 6.63 (d, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  (ppm): 169.52, 152.36, 145.57, 136.65, 135.74, 133.20, 131.55, 129.31, 128.17, 127.80, 123.19, 122.25, 119.00, 115.82; mass (m/z): 557 ( $M^+ + 1,6$ ), 556 ( $M^+$ , 10), 414 (10), 172 (20), 115 (23), 395 (40), 77 (70), 55 (100).

*1,1'-[4,4'-Diphenylsulfonebis(nitrilomethylidene)]bis-(2-naphthol) (3f)*. Orange solid, m.p. = 250-253 °C; IR (KBr),  $\nu/\text{cm}^{-1}$ : 3300-3550 (stretch, OH, H-bonded), 1613 (stretch, C=N); 1496-1588 (C=C, Ar), 1143 (stretch, C-O).  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  (ppm): 15.09 (s, 2O-H, intramolecular hydrogen bonding), 9.40 (s, CH imine), 7.74 (d, 2H), 7.69, 7.54 (dd, 8H), 7.58 (t, 2H), 7.17 (t, 2H), 6.78 (d, 2H), 6.63 (d, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  (ppm): 174.12, 155.46, 145.57, 139.12, 138.92, 136.75, 133.44, 132.91, 131.55, 129.56, 129.14, 128.15, 127.13, 123.25, 122.46; mass (m/z): 557 ( $M^+ + 1,6$ ), 556 ( $M^+$ , 10), 414 (10), 115 (23), 395 (40), 77 (70), 55 (100); anal. calcd.: 73.10 (C), 4.69 (H), 5.01 (N); found: 73.08(C), 4.71 (H), 5.03 (N).

*1,1'-[4,4'-Diphenylmethanebis(nitrilomethylidene)]bis-(2-naphthol) (3g)*. Reddish yellow solid, m.p. = 195-198 °C; IR (KBr),  $\nu/\text{cm}^{-1}$ : 3300-3500 (stretch, OH, H-bonded), 2922 (C-H, alkyl), 1623 (stretch, C=N), 1511-1544 (C=C, Ar), 1141 (stretch, C-O);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  (ppm): 15.65 (s, 2H, O-H, intramolecular hydrogen bonding), 9.40 (s, 2CH imine), 8.24 (d, 2H), 7.69 (2H, d), 7.57 (d, 2H), 7.38, 7.19 (8H, dd), 7.13 (t, 2H), 7.06 (t, 2H), 6.78 (d, 2H), 3.61 (s,  $\text{CH}_2$ ); mass (m/z): 509 ( $M^+ + 1$ , 4), 508 ( $M^+$ , 10), 145 (25), 103 (35), 170 (35), 76 (65), 220 (75), 262 (100); anal. calcd.: 82.96 (C), 5.17 (H), 5.53 (N); found: 82.94 (C), 5.16 (H), 5.58 (N).

*1,1'-[4-Hydroxy-2,6-pyrimidinediylbis(nitrilomethylidene)]bis-(2-naphthol) (3h)*. Yellow solid, m.p. = 251-253; IR (KBr),  $\nu/\text{cm}^{-1}$ : 3600-3500 (stretch, OH, H-bonded), 1614 (stretch, C=N), 1403-1486 (C=C, Ar), 1230 (stretch, C-O);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  (ppm): 11.15 (s, 2H, O-H, intermolecular hydrogen bonding), 9.34 (s, 2CH imine), 7.90 (m, 2H), 7.7 (d, 1H), 7.45 (d, 1H), 7.41 (d, 1H), 7.17-7.39 (m, 3H), 6.60-6.20 (m, 4H), 5.20 (s, OH); mass (m/z): 437 ( $M^+ + 1$ , 5), 436 ( $M^+$ , 10), 108 (16), 115 (20), 143 (35), 104 (50), 170 (55), 172 (75), 148 (100).

*1,1'-[2,6-Pyrimidinediyl-4-thiolbis(nitrilomethylidene)]bis-(2-naphthol) (3i)*. Yellow solid, m.p. = 276-279; IR (KBr),  $\nu/\text{cm}^{-1}$ : 3396 (stretch, OH), 1636 (stretch, C=N), 1466 (C=C, Ar), 1203 (stretch, C-O), 3128 (C-H aryl);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  (ppm): 11.05 (s, 2H, O-H, intermolecular hydrogen bonding), 9.22 (s, 2CH imine), 7.87 (m, 2H), 7.61 (d, 1H), 7.46 (d,

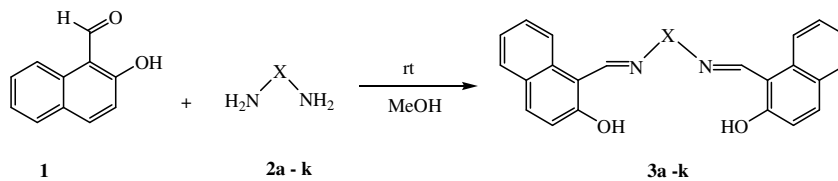
1H), 7.37 (1H, d), 7.09-7.31 (m, 3.5H), 6.65-6.12 (m, 4H). 2.20 (s, SH); mass (m/z): 451(M<sup>+</sup>+1, 4), 450 (M<sup>+</sup>, 12), 115 (15), 172 (25), 143 (35), 165 (40), 107 (47), 77 (78), 63 (90), 54 (100).

*1,1'-[4-Phenyl-2,6-diyl,3,5-triazinebis(nitrilomethylidene)]bis-(2-naphthol) (3j)*. Yellow solid, m.p. = 164-166 °C; IR (KBr),  $\nu/\text{cm}^{-1}$ : 3302 (stretch, OH), 1625 (stretch, (C=N), 1458-1541 (C=C, Ar), 1247 (stretch, C-O); <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$  (ppm): 13.92 (d, 2O-H, intermolecular hydrogen bonding), 9.50 (d, 2CH imine), 8.44 (d, 2H), 8.06 (d, 2H), 7.85-7.56 (m, 6H), 7.34 (t, 2H), 6.63 (d, 2H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz)  $\delta$  (ppm): 185, 171, 168, 163, 144, 142, 135, 133, 132, 130, 129.07, 129.8, 128, 126, 125, 120, 109; mass (m/z): 497 (M<sup>+</sup>+1, 6), 496 (M<sup>+</sup>, 10), 115 (20), 78 (27), 172 (30), 169 (40), 104 (50), 76 (85), 77 (100); anal. calcd.: 75.14 (C), 4.27 (H), 14.13 (N); found: 75.18 (C), 4.25 (H), 14.12 (N).

*1,1'-[2,4,6-Triyl-1,3,5-triazinebis(nitrilomethylidene)]tris-(2-naphthol) (3k)*. Yellow solid, m.p. = 150-153; IR (KBr),  $\nu/\text{cm}^{-1}$ : 3427 (stretch, OH), 1630 (stretch, C=N), 1403-1515 (C=C, Ar), 1247 (stretch, C-O); <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$  (ppm): 13.42 (s, 3O-H, intermolecular hydrogen bonding), 9.43 (s, 3CH imine), 8.93 (d, 3H), 8.33 (d, 3H), 7.99 (d, 3H), 7.74 (t, 3H), 6.86 (d, 3H); anal. calcd.: 73.46 (C), 4.11 (H), 14.28 (N); found: 73.48 (C), 4.12 (H), 14.27 (N).

## RESULTS AND DISCUSSION

The condensation reaction between 2-hydroxy-1-naphthaldehyde and various diamines in 2:1 molar ratio was obtained in high yields and short reaction times. The pure Schiff base as corresponding products were prepared in methanol solution at room temperature under mild reaction conditions (Scheme 1).



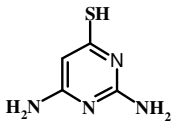
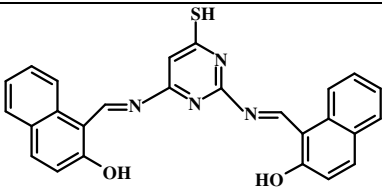
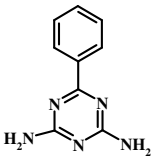
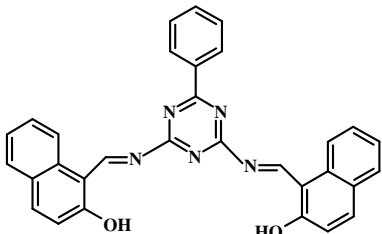
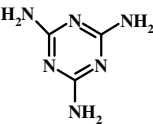
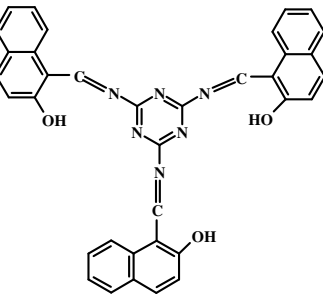
Scheme 1

When 2 mol of 2-hydroxy-1-naphthaldehyde, 1 mol various diamines were allowed to react at room temperature, and the corresponding products were characterized by spectroscopic and physical data. The results of these reactions are shown in Table 1. As indicated in the table, a lot of useful double Schiff bases were afforded in high yields and short reaction times from the one pot reaction of 2-hydroxy-1-naphthaldehyde with various diamines. The infrared spectrum of the Schiff base exhibited a band at 1625-1635  $\text{cm}^{-1}$  assignable to  $\nu(\text{C}=\text{N})$  of ligands.

The band of OH group in the ligands, the broad signal around  $\delta = 11.05$ -15.65 ppm are assigned to the protons of the hydroxyl groups. The protons of CH=N has the same chemical shifts in the range of  $\delta = 9.22$ -9.5 ppm. The signal around  $\delta = 6.0$ -6.65 ppm is assigned to the protons of the aromatic rings (CH=CH) and signals around  $\delta = 6.63$ -8.93 ppm. The signal around  $\delta = 155.76$ -185.17 ppm is assigned to the carbons of the NCHN and signals around  $\delta = 114$ -171 ppm are assigned to the carbons of aromatic rings (CH=CH).

Table 1. The obtained results related to the synthesis of Schiff bases.

Entry	Diamine	Product	Time (min)	Yield (%) <sup>a</sup>
1			3	95
2			8	91
3			10	90
4			45	92
5			30	89
6			90	75
7			100	72
8			80	74

9			100	77
10			90	65
11			90	50

<sup>a</sup>Isolated yield.

### CONCLUSION

The present work describes the synthesis of a new Schiff base ligand derived from 2-hydroxy-1-naphthaldehyde with various diamines. These ligands might have been interesting biological activity that would be specially designed research conducted by specialized biologists. In conclusion, we have reported a mild, easy, clean and a very efficient method for the synthesis of some novel Schiff bases without reflux conditions at room temperature. The products have been obtained in high yields and short reaction times. The structure of products has been assigned by physical and spectroscopic data such as; ultraviolet (UV-Vis), <sup>1</sup>H NMR, <sup>13</sup>C NMR, infrared (IR), elemental analysis (CHN) and mass spectra.

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