

Synthesis and Spectral Characterization of a Unsymmetrical Ligand, N,N'-Bis(4-fluorobenzylidene)propane-1,2-diamine and Its Zinc Complexes

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A new unsymmetrical bidentate Schiff base ligand *i.e.*, N,N'-bis(4-fluorobenzylidene)propane-1,2-diamine (BFBDP) was synthesized. Four zinc complexes of this ligand with general formula of Zn(BFBDP)X₂ in which X is Cl⁻, Br⁻, I⁻ or SCN⁻, have been prepared. The ligand and its complexes were characterized by elemental analyses, MS, ¹H NMR, ¹³C NMR, FT-IR and UV spectra and molar conductance. All compounds were found to be non-electrolytes in the DMF solutions.

Key Words: Schiff base, Zinc complex, Bidentate, Unsymmetrical.

INTRODUCTION

Schiff bases are known as biologically active compounds, liquid crystals, dyes, luminophores and polymer stabilizers¹⁻³. Antidepressants, antimicrobial, antitumor, antiphlogogistic, nematocide and other medicinal properties have been found as some new applications of these compounds^{4,5}. The role of the Schiff bases in the development of coordination chemistry providing the effects of steric interactions on coordination geometries is noteworthy⁶⁻¹¹. The widely studies have been done about the Schiff base metal complexes because of their industrial, antifungal, biological applications and other interesting properties of them¹²⁻¹⁴. Schiff bases and their biologically active complexes have been often used as radiopharmaceuticals for cancer targeting, agrochemicals, as model systems for biological macromolecules, as catalysts and as dioxygen carriers¹⁵⁻¹⁹. Although Schiff base metal complexes have been studied in sufficient details, but to the best of our knowledge the synthesis and spectral characterization of four coordinated zinc complexes of bidentate Schiff base ligand is new and scanty. In continuation our studies on four coordinated complexes²⁰⁻²³, in this work the synthesis, physical and spectral characterization of a new unsymmetrical bidentate Schiff base ligand, N,N'-bis(4-fluorobenzylidene)propane-1,2-diamine (BFBDP) and its zinc complexes with general formula of Zn(BFBDP)X₂ (X= Cl⁻, Br⁻, I⁻ or SCN⁻) (Fig. 1) are reported.

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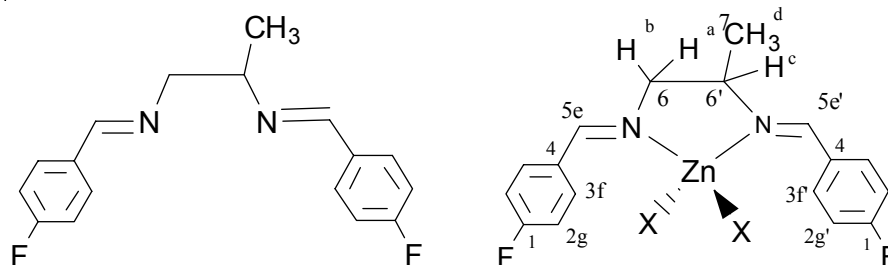


Fig. 1. Structural formula of ligand (BFBPD) and its zinc complexes

EXPERIMENTAL

4-Fluorobenzaldehyde, 1,2-propanediamine, zinc salts, solvent and other chemicals were purchased from either Aldrich, Merck or BDH Chemicals and were used without purification. IR spectra in the 4000-400 cm^{-1} range were obtained as KBr pellets on a JASCO-680 model FT-IR spectrometer. Electronic spectra were recorded in chloroform solutions on a JASCO-V570 model spectrometer. ^1H and ^{13}C NMR spectra were obtained using a Bruker DPX FT-NMR spectrometer at 500 MHz with the samples dissolved in CDCl_3 or $\text{CDCl}_3/\text{DMSO}-d_6$ mixture using TMS as internal standard. MS(m/z) of ligand was recorded on Shimadzu model GC-MS QP5050. Elemental analyses (CNHS) of complete dried samples were performed using a CHNS elemental analyzer by central instrumental laboratory of Tarbiat Moallem University of Tehran. The melting points ($^\circ\text{C}$) of the complexes were recorded on BI Barnstead electrothermal instrument. The molar conductivities of the ligand and their complexes were determined in DMF (1.0×10^{-3} M) at room temperature using Metrohm 712 conductometer with a dip-type conductivity cell made of platinum black.

Synthesis of Schiff base ligand (BFBPD): A solution of 4-fluorobenzaldehyde (0.496 g, 4 mmol) in absolute MeOH (10 mL) was added to a solution of 1,2-propanediamine, (0.15 g, 2 mmol) in absolute MeOH (10 mL) and severely stirred for 2-3 h. After completion of the reaction, the solvent was reduced under high ventilation vacuum to give the Schiff base ligand as the yellowish-white viscose oil. For purification, the oil obtained was washed twice with *n*-hexane as non-solvent and dried under vacuum to obtain the ligand with good purity with yield of 85 % (0.49 g).

Preparation of $\text{Zn}(\text{BFBPD})\text{X}_2$ ($\text{X} = \text{Cl}^-, \text{Br}^-, \text{I}^-$ or SCN^-) complexes: The zinc(II) complexes were prepared by stepwise addition and stirring of the ligand (0.143 g, 0.5 mmol) in methanol (10 mL) to the respective 0.5 mmol of zinc halide (ZnCl_2 (0.068 g), ZnBr_2 (0.113 g), ZnI_2 (0.159 g) and $\text{Zn}(\text{SCN})_2$ (0.5 mmol of it freshly was prepared in ethanolic solution²³) in methanol (30 mL) for 1-3 h. The white complexes, $\text{Zn}(\text{BFBPD})\text{X}_2$ so obtained were filtered, washed with EtOH twice and dried under vacuum to yield as in Table-1. The precipitates were further purified from dichloromethane/ethanol mixture and dried at (80-100 $^\circ\text{C}$) under vacuum and were kept in a desiccator over silica-gel.

RESULTS AND DISCUSSION

The BFBDP ligand was prepared by condensation of 4-fluorobenzaldehyde and 1,2-diaminopropane. It is soluble in dichloromethane, chloroform, acetone, alcohol, dimethyl sulfoxide, dimethyl formamide while is insoluble in *n*-hexane and *n*-heptane. The complexes were prepared by the reaction of the equimolar of the ligand and zinc salts. All isolated complexes are soluble in most organic solvents. The elemental analyses confirm 1:1 ratio of ligand to zinc salts for all complexes. The analytical data along with some physical properties of the complexes are summarized in Tables 1 and 2. The complexes can be formulated as Zn(BFBDP)X₂ (X = Cl⁻, Br⁻, I⁻ and SCN⁻). Molar conductivity values of 10⁻³ M solutions of the complexes in DMF at room temperature lies in the 15.37-102.22 cm² Ω⁻¹ M⁻¹ range which indicates that are all non-electrolytes and that the anion is also coordinated to the metal ion^{24,25}. Table-2 presents some important vibrational frequencies of the ligand BFBDP and its zinc complexes. Any vibrational frequencies assigned to the starting material including aldehyde and 1,2- propanediamine at 1688 and 3300-3100 cm⁻¹ were not observed in the IR spectrum of the ligand. The FT-IR spectrum of the Schiff base ligand of BFBDP showed the stretching frequencies at (3073, 3024), (2968, 2928) and 2848 cm⁻¹ assigned to C-H of aromatic, aliphatic and iminic groups, respectively. These frequencies are not affected considerably after coordination of the ligand. The frequencies at 1646 cm⁻¹ can be assigned to asymmetric vibration of the azomethine group (C=N)⁶⁻¹⁰. This band is shifted by 8-12 cm⁻¹ to lower frequencies in the spectra of the complexes, indicating coordination through the azomethine nitrogen of the Schiff base (Fig. 1). The stretching frequency of -C=N is shifted to lower frequencies after coordination is attributed to the π-back bonding of metal to ligand after σ-coordination of -C=N group^{9,24,26}.

TABLE-1
SOME ANALYTICAL DATA AND PHYSICAL PROPERTIES OF
THE LIGAND AND ITS ZINC COMPLEXES

| Compd. | m.w. (calcd.) | Color | m.p. (°C) | Yield (%) | Found (Calcd.) % | | | Λ _M (cm ² Ω ⁻¹ M ⁻¹) |
|------------------------------------|------------------|---------------------|----------------|--------------|------------------|----------------|------------------|--|
| | | | | | C | H | N | |
| BFBDP (1) | 286.32 | Light yellow | Viscose oil | 85 | 71.45 (71.31) | 5.71 (5.63) | 9.42 (9.78) | 1.92 |
| Zn(BFBDP)Cl ₂ (2) | 422.62 | White | 206 | 89 | 48.51 (48.31) | 3.69 (3.82) | 6.81 (6.63) | 15.37 |
| Zn(BFBDP)Br ₂ (3) | 511.52 | White | 256 | 79 | 39.81 (39.92) | 3.29 (3.15) | 5.61 (5.48) | 20.08 |
| Zn(BFBDP)I ₂ (4) | 605.52 | Yellowish- white | 260 | 85 | 34.58 (33.72) | 2.57 (2.66) | 4.51 (4.63) | 102.20 |
| Zn(BFBDP)(SCN) ₂ (5) | 467.87 | White | 227 | 75 | 48.58 (48.77) | 3.57 (3.45) | 11.79 (11.97) | 43.75 |

The frequencies at 1509, 1449 and 1415 cm^{-1} show C=C stretching vibrations. The bending vibration of methyl groups is appeared at 1380 cm^{-1} . The vibrational frequencies of (C-F) in the compounds are observed at 1236-1230 cm^{-1} . The strong out of plane bending vibration of the aromatic C-H is present²⁷ at 834 cm^{-1} . This band is nearly unchanged after the coordination. In the spectra of the zinc complexes, the bands observed in the 510-481 cm^{-1} regions that are absent in the ligand spectrum may be due to symmetrical and unsymmetrical vibration mode of (M-N)¹⁸, suggesting that the complexes are four coordinate with two azomethine nitrogens of bidentate ligand and two halides. The new intense bands at 2072 and 2060 cm^{-1} in the IR spectrum of Zn(BFBPD)(SCN)₂ is safely attributed to N- coordinated⁷ SCN⁻. The electronic absorption data of the compounds, which were measured in DMF, are given in Table-2. In the spectrum of the ligand, three bands at 247, 278 (shoulder) and 287 (shoulder) nm are seen. During the formation of the complexes, these bands may be shifted to lower or higher wavelengths, suggesting that the nitrogen

TABLE-2
FT-IR (cm^{-1}) AND UV-VIS (nm) SPECTRAL DATA OF THE
SCHIFF BASE (BFBPD) AND ITS ZINC COMPLEXES

| Compd. | $\nu\text{CH}_{\text{arom.}}$ | $\nu\text{CH}_{\text{aliph.}}$ | $\nu\text{CH}_{\text{imin.}}$ | $\nu_{\text{asym}}(\text{C}=\text{N})$ | $\nu_{\text{sym}}(\text{C}=\text{N})$ | $\nu(\text{C}=\text{C})$ |
|----------|-------------------------------|-----------------------------------|--------------------------------------|--|---------------------------------------|---|
| 1 | 3073(w), 3024(w) | 2968(m), 2928(m) | 2848(m) | 1646(s) | 1602(s) | 1508(s) 1449(m), 1415(m), |
| 2 | 3109(w), 3073(w) | 2974(w), 2933(w), 2912 | 2852(w) | 1638(vs) | 1600(vs) | 1509(s), 1446(s), 1428(m), |
| 3 | 3109(w), 3068(w) | 2973(w), 2921(w) | 2852(w) | 1638(vs) | 1600(vs) | 1509(s), 1437(m), 1397(m) |
| 4 | 3105(w), 3063(w) | 2964(w), 2937(w), 2924(w) | 2862(w) | 1634(vs) | 1600(vs) | 1509(s), 1456(m), 1440(m) |
| 5 | 3106(w), 3073(w) | 2971(w), 2925(w) | 2861(w) | 1637(vs) | 1600(vs) | 1509(vs) 1445(s), 1428(w), |
| | $\nu(\text{C-F})$ | $\delta\text{CH}_{\text{methyl}}$ | $\delta\text{CH}_{\text{arom(oop)}}$ | $\nu(\text{Zn-N})$ | $\nu(\text{SCN})$ | λ_{max} ($\text{C}, \text{cm}^{-1}\text{M}^{-1}$) |
| 1 | 1230(s) | 1380(w) | 834(vs) | - | - | 247(41693) 278(sh), 287(sh) |
| 2 | 1235(vs) | 1383(m) | 836(vs) | 504(m), 481(m) | - | 264(7624) 277(sh), 287(sh) |
| 3 | 1233(vs) | 1383(m) | 837(vs) | 502(m), 481(w) | - | 264(6044) 277(sh), 286 |
| 4 | 1231(vs) | 1378(m) | 834(vs) | 498(m), 477(m) | - | 264(6273), 280(sh), 289(sh) |
| 5 | 1236(vs) | 1346(m) | 837(vs) | 510(m), 483(m) | 2072(vs), 2060(vs) | 264(6942) 278(sh), 286(sh) |

atom of the azomethine group is coordinated to the metal ion. The first two values at 247-280 nm region of the spectra are attributed to the π - π^* transition of aromatic rings in the ligand structure. The third electronic absorption band of the ligand and Zn(II) complex as shoulder at the 286-290 nm may be due to the π - π^* transitions of the azomethine group which is mainly localized within the imine chromophore. The d - d transition bands are not observed for zinc complexes. The electronic spectra of zinc complexes generally consists charge transfer (L-M) transition that in titled complexes may be overlapped with π - π^* transition of the ligand⁷. The suggested structure for the zinc complexes with considering our previous report on this type of ligands²⁰⁻²³ is pseudo-tetrahedral as drawn in Fig. 1. Mass spectral data of the Schiff base ligand includes main fragments of 286 (M⁺), 268 (M-F), 252, 248 (M-2F), 235, 233, 219, 217, 164, 148, 122, 112, 97, 85, 79, 70, 57, 51 (m/z). Electron impact (EI) mass spectroscopy (MS) lends support to the formula shown in Fig. 1. The mass spectrum of the ligand presents the peak corresponding to the molecular ion 286 (M⁺) that indicates stability of ligand molecules in gas phase. Other fragments are in agreement with the molecular structure of the ligand. The NMR spectra of the ligand and its zinc complexes were recorded using CDCl₃ or CDCl₃/DMSO mixture as solvent at 500 MHz and their data were summarized in Tables 3 and 4.

TABLE-3
¹H NMR CHEMICAL SHIFTS OF LIGAND AND ITS ZINC COMPLEXES IN ppm
 IN CDCl₃ OR CDCl₃/DMSO MIXTURE AS SOLVENT

| Compd. ^b | Assignment of the protons |
|---------------------|---|
| 1 | 8.26 (s, 1H _e), 8.21 (s, 1H _{e'}), 7.70 (t, 2H _g , J _{HF} = 8.86 Hz and J _{HH} = 8.86 Hz), 7.69 (t, 2H _g , J _{HF} = 9.23 Hz and J _{HH} = 8.83 Hz), 7.07 (t, 4H(2H _f , 2H _f), J _{HF} = 8.85 Hz and J _{HH} = 8.85 Hz), 3.89 (dd, 1H _b , J = 10.58 Hz and J = 4.25 Hz), 3.77 (sextet, 1H _c , J = 5.60 Hz), 3.73 (dd, 1H _a , J = 10.30 Hz and J = 7.45 Hz) and 1.39 (d, 3H _d , J = 6.14 Hz). |
| 2 | 8.30 (s, 1H _e), 8.27 (s, 1H _{e'}), 7.77 (bs, 4H(2H _g , 2H _g)), 6.98 (t, 4H(2H _f , 2H _f), J _{HF} = 8.40 Hz and J _{HH} = 8.38 Hz), 3.91 (bd, 1H _b , J = 11.65 Hz), 3.83 (bs, 1H _c), 3.58 (dd, 1H _a , J = 12.46 Hz and J = 4.61 Hz) and 1.23 (d, 3H _d , J = 6.58 Hz). |
| 3 | 8.38 (s, 1H _e), 8.36 (s, 1H _{e'}), 7.83 (bs, 4H(2H _g , 2H _g)), 7.06 (t, 4H(2H _f , 2H _f), J _{HF} = 8.12 Hz and J _{HH} = 8.12 Hz), 4.02 (bs, 1H _b), 3.93 (bs, 1H _c), 3.64 (dd, 1H _a , J = 12.57 Hz and J = 4.26 Hz) and 1.32 (d, 3H _d , J = 6.56 Hz). |
| 4 | 8.61 (s, 1H _e), 8.59 (s, 1H _{e'}), 8.26 (t, 2H _g , J = 9.40 Hz and J _{HH} = 9.40 Hz), 7.69 (t, 2H _g , J _{HF} = 9.39 Hz and J _{HH} = 9.39 Hz), 7.27 (t, 4H(2H _f , 2H _f), J _{HF} = 8.56 Hz and J _{HH} = 8.55 Hz), 4.42 (d, 1H _b , J = 13.15 Hz), 4.24 (m, 1H _c), 3.73 (dd, 1H _a , J = 13.26 Hz and J = 1.94 Hz) and 1.57 (d, 3H _d , J = 6.86 Hz). |
| 5 ^a | 8.23 (s, 1H _e), 8.19 (s, 1H _{e'}), 7.59 (t, 2H _g , J = 5.55 Hz and J _{HH} = 5.52 Hz), 7.57 (t, 2H _g , J _{HF} = 5.53 Hz and J _{HH} = 5.55 Hz), 6.96 (t, 4H(2H _f , 2H _f), J _{HF} = 8.51 Hz and J _{HH} = 8.58 Hz), 3.77 (dd, 1H _b , J = 11.78 Hz and J = 4.60 Hz), 3.72 (sextet, 1H _c , J = 5.98 Hz), 3.57 (dd, 1H _a , J = 11.83 Hz and J = 5.84 Hz) and 1.17 (d, 3H _d , J = 6.40 Hz). |

^aThe ¹H NMR of the complex, **2**, **3** and **5** was recorded in DMSO/CDCl₃ mixture.

TABLE-4
¹³C NMR CHEMICAL SHIFTS OF THE LIGAND AND ITS ZINC
 COMPLEXES IN ppm IN CDCl₃ SOLVENT

| Compd. | Assignment of the protons |
|----------------------|--|
| 1 | 165.62(C1', d, <i>J</i> = 18.5 Hz), 163.62(C1, d, <i>J</i> = 18.0 Hz), 161.41(C5'), 159.52(C5), 132.99(C3', d, <i>J</i> = 11.5 Hz), 132.91(C3, d, <i>J</i> = 12.0 Hz), 130.37(C2', d, <i>J</i> = 25.5 Hz), 130.31(C2, d, <i>J</i> = 26.0 Hz), 116.11(C4', d, <i>J</i> = 23.5 Hz), 115.91(C4 d, <i>J</i> = 24.0 Hz), 77.53(t, CDCl ₃), 68.04(C6'), 66.72(C6), 20.92(C7). |
| 2 | 166.28(C1'), 164.31(C1), -(C5'), -(C5), 132.55(C3'), 131.97(C3), -(C2'), -(C2), 116.37(C4', d, <i>J</i> = 16.5 Hz), 116.18(C4 d, <i>J</i> = 16.5 Hz), 77.82(t, CDCl ₃), 66.39(C6'), 65.52(C6), 40.33(septet, DMSO-d ₆), 20.63(C7) |
| 4 | 169.67(C5'), 168.28(C5), 167.55(C1', d, <i>J</i> = 11.72 Hz), 165.50(C1, d, <i>J</i> = 11.80 Hz), 135.00(C3', d, <i>J</i> = 12.0 Hz), 134.92(C3, d, <i>J</i> = 13.0 Hz), 127.80(C2', d, <i>J</i> = 11.23 Hz), 127.70(C2, d, <i>J</i> = 11.28 Hz), 116.84(C4', d, <i>J</i> = 14.0 Hz), 116.65(C4 d, <i>J</i> = 14.0 Hz), 77.46(t, CDCl ₃), 65.68(C6'), 65.32(C6), 21.12(C7) |
| 5^a | 165.96(C1', d, <i>J</i> = 30.0 Hz), 164.31(C5'), 163.95(C1, d, <i>J</i> = 29.0 Hz), 162.25(C5), 137.68(C _{SCN}), 131.54(C3'), 131.39(C3), 130.94(C2'), 130.87(C2), 116.34(C4', d, <i>J</i> = 28.0 Hz), 116.17(C4 d, <i>J</i> = 28.0 Hz), 77.84(t, CDCl ₃), 66.90(C6'), 65.78(C6), 40.42(septet, DMSO-d ₆), 20.58(C7) |

^aThe ¹³C NMR of the complex, **2** and **5** was recorded in DMSO/CDCl₃ mixture.

The ¹H and ¹³C NMR spectral studies of the ligand and its zinc complexes strongly support the proposed geometry of the compounds. The ¹H NMR spectrum of the ligand includes the azomethine protons (H_e and H_{e'}) resonances as functional group signals at 8.26-8.21 ppm⁷. These signals are red shifted to 8.61-8.27 ppm in its zinc(II) complexes with respect to TMS (except for complex **5** that shifted to 8.19-8.23 ppm), suggesting well coordination by the azomethine nitrogens to metal ion. It is interesting to note that the protons, H_a, H_b and H_c of the ligand and its zinc complexes are clearly distinguished. In the spectrum of the ligand, H_a is observed at 3.73 ppm as doublet of doublet due to coupling with the H_c and H_b, respectively. H_b is appeared similar to H_a at 3.89 ppm as doublet of doublet because coupling with H_a and H_c, respectively. In the complexes H_b is appeared as broad doublet and broad singlet at weaker fields except for **5** that appeared similar to the one of the ligand at higher field. H_a is stayed doublet of doublet in all complexes and are shifted to higher fields after coordination. The signal of the H_c in the ligand spectrum is appeared as clear sextet because of its coupling with H_d and then with H_a and H_b with coupling constant of 5.60 Hz. But this signal is observed as broad singlet or multiplet except for **5** with notable shifts to lower field after coordination. H_d of the ligand is observed as a doublet at 1.39 ppm due to coupling with H_c that shifted to 1.17-1.32 ppm in the complexes, **2**, **3** and **5** while it shifted to 1.57 ppm in the spectrum of **4**. H_g and H_{g'} signals of the ligand are seen as triplet at 7.70-7.69 ppm with J_{HF} and J_{HH} (8.86 Hz) that shifted to weaker fields in the complexes of **2**, **3** and **4** but to stronger fields for the complex **5**. These observations for H_d, H_g and H_{g'} may be related to more effective π-back bonding of metal to ligand in the complexes

that is affected by coordinated halides and/or thiocyanate. The ¹³C NMR spectrum of the ligand shows two doublet peaks at 165.62 and 163.62 ppm for C(1', 1) carbons due to coupling with fluorine atom with $J = 18.5$ and 18.0 Hz, respectively that shifted to lower energies after coordination. The coupling effect of fluorine atom are continued until C(4', 4) of benzene ring. Two azomethine carbons C(5',5) resonances of the ligand are observed at 161.41 and 159.52 ppm. These peaks are red shifted in its zinc(II) complexes such as **4** and **5**, suggesting well coordination of the azomethine nitrogen to metal ion. Among the other signals, C(2',2), C(3',3), C(6',6) and C(7',7) are blue shifted but C(4',4) red shifted after coordination of the ligand. These changes in ¹³C NMR chemical shifts are rationalized based on the probable resonance during the conjugation system of the free ligand and limitation of it as well as π -back bonding of metal to ligand after coordination. Some carbon atoms of the compounds **2** and **3** were not appeared even after 10 h in the NMR instrument probably due to not-enough solubility of it in solvents.

Conclusion

In this work, the synthesis and spectral characterization of a new unsymmetrical bidentate Schiff base ligand and its zinc complexes are described. The results supported 1:1 ratio of ligand to zinc salts.

Supplementary

The Mass spectrum of the ligand, ¹H NMR and ¹³C NMR of the ligand and its complexes are found at the supplementary file.

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