

## Supramolecular Interactions Involved in the Solid State Structure of *N,N'*-[bis(pyridin-2-yl)formylidene]ethane-1,2-diamine

Arab K. El-Qisairi,<sup>a,\*</sup> Hanan A. Qaseer,<sup>a</sup> Solhe F. Alshahateet,<sup>a</sup> M. K. Hasan Qaseer,<sup>b</sup> Mukarram H. Zaghal,<sup>c</sup> Wa'el Al-Btoush,<sup>a</sup> and Louise N. Dawe<sup>d</sup>

<sup>a</sup>Department of Chemistry, Faculty of Science, Mu'tah University, Mu'tah 61710, Jordan

<sup>b</sup>Department of Physics, Faculty of Science, Jordan University of Science and Technology, P.O.Box 3030, Irbid, Jordan

<sup>c</sup>Department of Chemistry, Faculty of Science, Yarmouk University, Irbid, Jordan

<sup>d</sup>Centre for Chemical Analysis, Research and Training, Memorial University of Newfoundland St. John's, NL, A1B 3X7, Canada

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**Abstract.** The structure of the symmetrical Schiff base, *N,N'*-[bis(pyridin-2-yl)formylidene]ethane-1,2-diamine (bpdf) has been characterized by single crystal X-ray diffraction. The non-covalent supramolecular chemistry involved in the crystal structure of this ligand has been carefully investigated. The structure adopted different motifs of nitrogen-hydrogen interactions that led to the formation of centrosymmetric dimers. In addition, edge-edge and face-face nitrogen-nitrogen interactions were observed and reported. The Schiff base (bpdf) ligand crystallizes in a monoclinic space group *C*12/c1 with  $a = 19.128(2)$  Å;  $b = 5.8776(6)$  Å;  $c = 13.1403(15)$  Å;  $\alpha = 90^\circ$ ;  $\beta = 121.970^\circ(4)$ ;  $\gamma = 90^\circ$  and  $z = 4$ . This structure is an example of compounds with many symmetry-independent molecules in the asymmetric unit cell ( $Z > 2$ ).

**Keywords:** centrosymmetric dimer interactions, unit cell packing arrangement, spectral properties, high *Z*-value structure

### INTRODUCTION

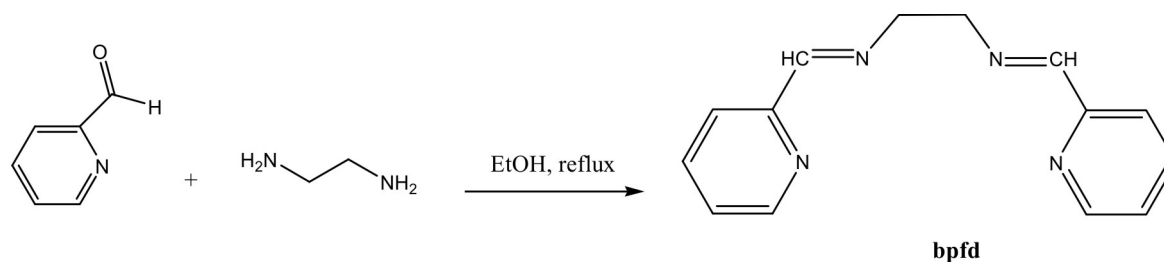
Recently, there has been some interest in the preparation of various Schiff base ligands<sup>1</sup> due to their preparative accessibilities, structural varieties and varied denticities. Metal-chelate Schiff base complexes have played an important role in developing stereochemical models in main group and transition metal coordination chemistry, mainly due to their stability, ease of preparation, and structural variability.<sup>2–6</sup> Furthermore, they have been investigated for their ability to act as antibacterial agents,<sup>7–9</sup> antifungal agents,<sup>10,11</sup> antitumor drugs<sup>12–14</sup> and catalysts.<sup>15–17</sup> The *N,N'*-[bis(pyridin-2-yl)formylidene]ethane-1,2-diamine (bpdf) ligand is among the ligands that have been studied and some of its complexes have been reported.<sup>1,18–23</sup> As a part of our ongoing research into the structure and utility of Schiff base ligands, we report the crystal structure of *N,N'*-[bis(pyridin-2-yl)formylidene]ethane-1,2-diamine (bpdf) ligand, and study the supramolecular chemistry is based on non-covalent interactions involved in the crystal structure of (bpdf) ligand.

### EXPERIMENTAL

#### General Experimental

All chemicals were purchased from Aldrich chemical company and used as received. The solvents used were AR grade. <sup>1</sup>H and <sup>13</sup>C NMR spectra were recorded in CDCl<sub>3</sub> on a Bruker 400 MHz at Jordan University of Science and Technology. Chemical shifts were referenced to tetramethylsilane (TMS). The infrared spectra were recorded on a Maltson 5000 FTIR spectrophotometer. Electronic absorption spectra were recorded on a Perkin Elmer Lambda 25 UV/Vis Spectrometer in dimethylsulfoxide (DMSO) using 1 × 10<sup>-6</sup> M solution. The symmetrical Schiff base, *N,N'*-[bis(pyridin-2-yl)formylidene]ethane-1,2-diamine (bpdf) was prepared by refluxing ethane-1,2-diamine and pyridine-2-carboxaldehyde in 1:2 mole ratio in absolute ethanol (Figure 1).<sup>24</sup> The (bpdf) ligand was characterized by FTIR, UV-vis, <sup>1</sup>H and <sup>13</sup>C NMR spectroscopy. It was recrystallised from diethyl ether to give colourless crystals with X-ray quality.

\* Author to whom correspondence should be addressed. (E-mail: aqaseer@mutah.edu.jo & aqaseer2011@yahoo.com)



**Figure 1.** Preparation of bpdf ligand

### Preparation of C<sub>14</sub>H<sub>14</sub>N<sub>4</sub> (bpdf)

To a stirred solution of ethylenediamine (0.157 g; 2.61 mmol) in 20 ml ethanol, a solution of 2-pyridinecarboxaldehyde (0.560 g; 5.23 mmol) in ethanol (5 ml) was added dropwise over a period of 1–2 hours. The reaction mixture was then stirred and refluxed for 24 hours. The mixture was evaporated and recrystallized from diethyl ether. The product was filtered and dried in oven at 40 °C. The yield was 0.30 g (48 %). The compound melted at 57–59 °C. FTIR (neat, cm<sup>-1</sup>): 2920  $\nu$ (C–H, aliphatic), 1647  $\nu$ (C=N, imine), 1586, and 1427  $\nu$ (C=N, and C=C pyridine). UV-vis;  $\lambda_{\max}(\epsilon)$ : 272 nm (22900 M<sup>-1</sup> cm<sup>-1</sup>), 282 (15500 M<sup>-1</sup> cm<sup>-1</sup>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  = 8.59 (d, 2H,  $J$  = 6.1), 8.40 (s, 2H), 7.95 (d, 2H,  $J$  = 6.1), 7.69 (t, 2H,  $J$  = 7.6), 7.25 (dt, 2H,  $J$  = 1.6, 6.1), 4.03 (s, 4H) ppm. <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):  $\delta$  = 61.3, 121.3, 124.4, 136.5, 149.3, 154.3, 163.2 ppm. The solid state structure of the (bpdf) ligand has been determined by single-crystal X-ray diffraction.

### Crystal Data

Empirical Formula	C <sub>14</sub> H <sub>14</sub> N <sub>4</sub>
Formula Weight	238.29 g/mol
Crystal Color, Habit	chunk, colorless
Crystal Dimensions	0.80 X 0.80 X 0.78 mm
Crystal System	monoclinic
Lattice Type	C-centered
Detector Position	40.03 mm
Pixel Size	0.137 mm
Lattice Parameters	$a = 19.128(2)$ Å $b = 5.8776(6)$ Å $c = 13.1403(15)$ Å $\beta = 121.970(4)^\circ$ $V = 1253.3(2)$ Å <sup>3</sup>
Space Group	C12/c1
Z value	4

$D_{\text{calc}}$  1.263 g/cm<sup>3</sup>

$F_{000}$  504

$\mu(\text{MoK}\alpha)$  0.79 cm<sup>-1</sup>

### Intensity Measurements

Detector	Rigaku Saturn
Goniometer	Rigaku AFC8
Radiation	MoK $\alpha$ ( $\lambda = 0.71070$ Å) graphite monochromated
Detector Aperture	70 mm × 70 mm
Data Images	864 exposures
$\omega$ oscillation Range ( $\chi = 0.0, \phi = 0.0$ )	-75.0–105.0°
Exposure Rate	22.0 sec./°
Detector Swing Angle	15.08°
$\omega$ oscillation Range ( $\chi = 0.0, \phi = 180.0$ )	-45.0–75.0°
Exposure Rate	22.0 sec./°
Detector Swing Angle	15.08°
$\omega$ oscillation Range ( $\chi = 45.0, \phi = 90.0$ )	-42.0–90.0°
Exposure Rate	22.0 sec./°
Detector Swing Angle	15.08°
Detector Position	40.00 mm
Pixel Size	0.068 mm
$2\theta_{\text{max}}$	80.6°
No. of Reflections Measured	Total: 2592 Unique: 1420 ( $R_{\text{int}} = 0.0214$ )
Corrections	Lorentz-polarization Secondary Extinction (coefficient: 0.0066(16)) Absorption (trans. factors: 0.9394–0.9408)

**Structure Solution and Refinement**

Structure Solution	Direct Methods (SHELX97)
Refinement	Full-matrix least-squares on $F^2$
Function Minimized	$\Sigma w (F_o^2 - F_c^2)^2$
Least Squares Weights	$w = 1 / [ \sigma^2(F_o^2) + (0.0202 \cdot P)^2 + 1.2836 \cdot P ]$ where $P = (\text{Max}(F_o^2, 0) + 2F_c^2)/3$
$2\theta_{\text{max}}$ cut off	55.0°
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	1420
No. Variables	84
Reflection/Parameter Ratio	16.90
Residuals: $R1$ ( $I > 2.00 \sigma(I)$ )	0.0458
Residuals: $R$ (All reflections)	0.0482
Residuals: $wR2$ (All reflections)	0.0944
Goodness of Fit Indicator	1.223
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	$0.29 \text{ e}^-/\text{\AA}^3$
Minimum peak in Final Diff. Map	$-0.17 \text{ e}^-/\text{\AA}^3$

**Solution and Refinement of the Crystal Structure**

Single-crystal X-ray diffraction experiment was carried out on a Rigaku Saturn CCD area detector with graphite monochromate Mo-K $\alpha$  radiation. The data were collected at a temperature of  $-135 \pm 1$  °C to a maximum  $2\theta$  value of 80.6°. Data were collected and processed using Crystal Clear.<sup>25</sup> The linear absorption coefficient,  $\mu$ , for Mo-K $\alpha$  radiation is  $0.79 \text{ cm}^{-1}$ . A numerical absorption correction was applied which resulted in transmission factors ranging from 0.9394 to 0.9408. The data were corrected for Lorentz and polarization effects.<sup>26</sup> The structure was solved by direct methods<sup>27</sup> and expanded using Fourier techniques.<sup>28</sup> The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement<sup>29</sup> on  $F^2$  was based on 1420 observed reflections and 84 variable parameters and converged. Crystallographic data for the structural analysis reported in this paper have been deposited with the Cambridge Crystallographic Data Centre, CCDC, Number (721559). This data can be obtained free of charge at <http://www.ccdc.cam.ac.uk/conts/retrieving.html> [or from the Cambridge Crystallographic Data Centre

(CCDC), 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44-1223-336033; email: deposit@ccdc.cam.ac.uk].

Neutral atom scattering factors were taken from Cromer and Waber.<sup>30</sup> Anomalous dispersion effects were included in  $F_{\text{calc}}$ ,<sup>31</sup> the values for  $\Delta f'$  and  $\Delta f''$  were those of Creagh and McAuley.<sup>32</sup> The values for the mass attenuation coefficients are those of Creagh and Hubbell.<sup>33</sup> All calculations were performed using the CrystalStructure<sup>25,34</sup> crystallographic software package except for refinement, which was performed using SHELXL-97.<sup>29</sup>

**RESULTS AND DISCUSSION****IR and UV/vis Spectroscopy**

The Schiff base (bpdf) ligand has a strong absorbance at  $1647 \text{ cm}^{-1}$  in the infrared region, confirming the presence of the imine moiety. The stretching vibration of C=N and C=C of the pyridine ring are observed at 1586 and  $1427 \text{ cm}^{-1}$ , respectively. Additionally, the UV/vis spectrum has strong absorptions near 272 and 282 nm. These bands are assigned to the ligand centered (LC) or  $\pi$ - $\pi^*$  transitions. The IR data are consistent with the reported value.<sup>35</sup>

**NMR Spectroscopy**

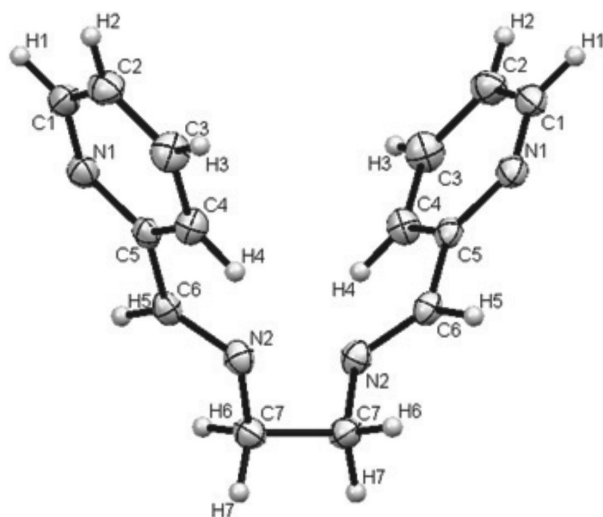
The  $^1\text{H}$  NMR spectrum for the (bpdf) ligand give signals corresponding to the aromatic protons in the range 7.25–8.59 ppm. The  $^1\text{H}$  NMR signals arising from ethylene protons appear as singlet at 4.02 ppm.  $^{13}\text{C}$  NMR spectrum supports the formation of the Schiff base (bpdf) ligand. The  $^{13}\text{C}$  NMR signals corresponding to the aromatic carbons are visible in the region 117–166 ppm. Signal corresponding to the aliphatic and vinylic carbons are found at 61 and 154 ppm, respectively.

**Supramolecular Interactions Involved in the Solid State Structure of (bpdf) Ligand**

A solvent free sample of the *N,N'*-[bis(pyridine-2-yl)-formylidene] ethane-1,2-diamine ligand (bpdf) (Figure 1) was obtained after crystallization from diethyl ether to give material that was good enough to be examined by the single crystal X-ray technique. The (bpdf) ligand crystallizes in a monoclinic space group C12/c1 with  $a = 19.128(2) \text{ \AA}$ ;  $b = 5.8776(6) \text{ \AA}$ ;  $c = 13.1403(15) \text{ \AA}$ ;  $\alpha = 90^\circ$ ;  $\beta = 121.970^\circ(4)$ ;  $\gamma = 90^\circ$  and  $z = 4$ . Numerical details of the crystal structure are presented in experimental section. ORTEP drawing of (bpdf) molecular structure is shown in Figure 2. The bond distances and angles for (bpdf) ligand (Tables 1, 2) are as expected.<sup>36,37</sup> The crystal structure of (bpdf) is an example of compounds with many symmetry-independent mole-

**Table 1.** Summary of bond distances (Å) for (bpfd) ligand

atom	atom	distance	atom	atom	distance
N(1)	C(1)	1.3459(18)	N(1)	C(5)	1.3547(18)
N(2)	C(6)	1.2715(16)	N(2)	C(7)	1.4666(18)
C(1)	C(2)	1.396(2)	C(2)	C(3)	1.391(2)
C(3)	C(4)	1.394(2)	C(4)	C(5)	1.400(2)
C(5)	C(6)	1.4870(18)	C(7)	C(7)	1.5272(14)

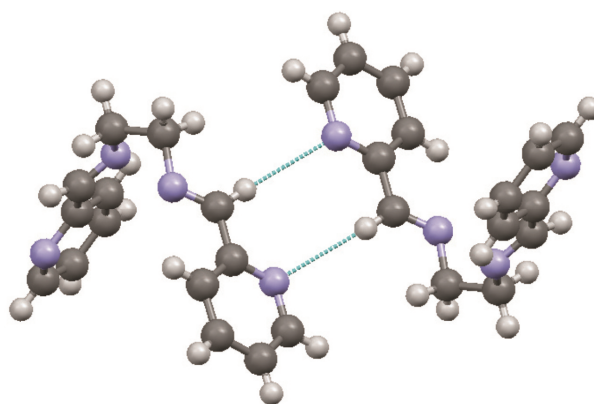
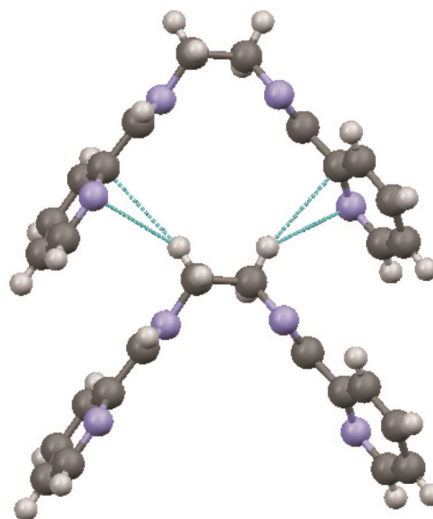
**Figure 2.** ORTEP drawing of (bpfd). Thermal ellipsoids are shown in 50 % probability.

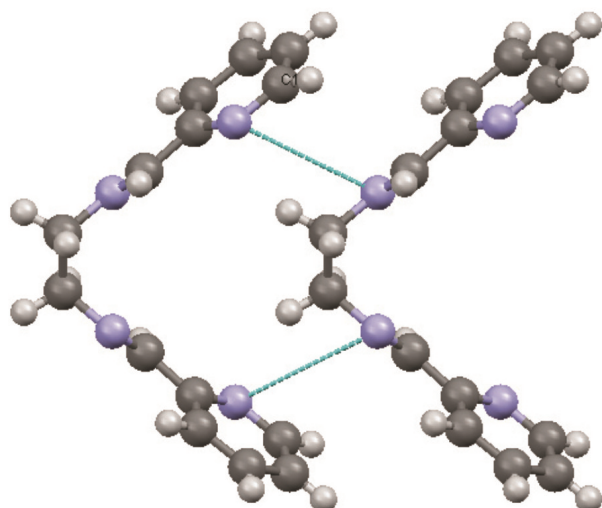
cules in the asymmetric unit cell ( $Z > 2$ ). This phenomenon was extensively analyzed by Bernstein *et al.*<sup>38</sup> and accordingly reported that high- $Z$  structures have been described as “snapshot pictures” and also as “fossil relics” of early stages in crystallization. Such description has a certain charm. However, as we are almost totally ignorant of the nucleation process, the early stages in crystallization, these picturesque descriptions have the disadvantage that they are almost impossible to confirm or to refute.

**Table 2.** Summary of bond angles (°) for (bpfd) ligand

atom	atom	atom	angle
C(1)	N(1)	C(5)	117.22(14)
N(1)	C(1)	C(2)	123.44(13)
C(2)	C(3)	C(4)	119.27(17)
N(1)	C(5)	C(4)	123.33(12)
C(4)	C(5)	C(6)	121.60(12)
N(2)	C(7)	C(7)	111.46(11)
C(6)	N(2)	C(7)	116.99(13)
C(1)	C(2)	C(3)	118.56(13)
C(3)	C(4)	C(5)	118.16(14)
N(1)	C(5)	C(6)	115.07(13)
N(2)	C(6)	C(5)	121.82(14)

Detailed analysis of (bpfd) crystal structure showed the presence of edge-edge C6–H5...N1 centrosymmetric dimer interactions. This centrosymmetric C–H...N dimer is composed of  $\overline{C-H...N}$  and  $\underline{C-H...N}$  interactions with distances of 2.7 and 3.55 Å, respec-

**Figure 3.** Centrosymmetric dimer composed of two molecules of (bpfd) through C6–H5...N1 interactions with a bond distance of 2.7 Å and bond angle of 145.68°.**Figure 4.** Part of ligand (bpfd) crystal structure showing bifurcated (N1...H7...C5) centrosymmetric dimers formed between methylene hydrogen and pyridine nitrogen with a distance of 2.73 Å and pyridine carbon and methylene hydrogen with a distance of 2.84 Å and N1...H7...C5 bond angle of 28.09°.



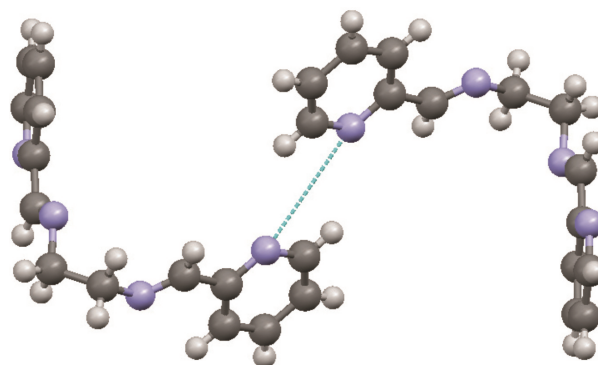
**Figure 5.** Two molecules of (bpdf) ligand interact together through their nitrogen atoms to form centrosymmetric dimer with a distance of 3.92 Å.

tively and C6–H5...N1 bond angle of 145.68° as shown in Figure 3.

In addition, detailed analysis of (bpdf) crystal structure showed the presence of a layer arrangement between the aromatic ring and aliphatic methyl hydrogen to produce the bifurcated centrosymmetric C–H...N and C–H...C interactions with distances of 2.73 and 2.84 Å, respectively as shown in (Figure 4). In addition, face-face nitrogen-nitrogen interaction is observed in the crystal structure of (bpdf) through C1–N1...N2 to form centrosymmetric dimer. The centrosymmetric C1–N1...N2 dimer had C–N...N interactions with a distance of 3.92 Å and bond angle of 63.92° as shown in Figure 5. This centrosymmetric dimer is stabilized by mean of face-face  $\pi$ -stacking interactions between the pyridine rings and the C1–N1 of each dimer with distances of 3.92 Å with offset angle of 63.92° (Figure 5). Such interactions will stabilize the formation of the dimer. Furthermore, edge-edge nitrogen-nitrogen interaction is observed between two molecules of (bpdf) through C1–N1...N1–C1 interaction with a bond distance of 3.89 Å and C1–N1...N1 bond angle of 68.79° as shown in Figure 6.

## CONCLUSION

The structure of the Schiff base, *N,N'*-[bis(pyridine-2-yl)formylidene]ethane-1,2-diamine (bpdf) has been characterized by NMR spectroscopy and a single-crystal X-ray diffraction. The non-covalent supramolecular chemistry involved in the crystal structure of this ligand has been carefully investigated. The analysis of bpdf ligand crystal structure demonstrates the presence of different supramolecular synthons such as edge-edge (aliph-



**Figure 6.** Edge-edge nitrogen-nitrogen interaction between two molecules of (bpdf) ligand with a distance of 3.89 Å.

H...N1 and C1–N1...N1–C1) and face-face C1–N1...N2 centrosymmetric dimer interactions. The crystal structure of (bpdf) is found to be a new example of compounds with many symmetry-independent molecules in the asymmetric unit cell ( $Z > 2$ ). Different motifs of non-covalent interactions were observed and reported in terms of crystal engineering and supramolecular chemistry.

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