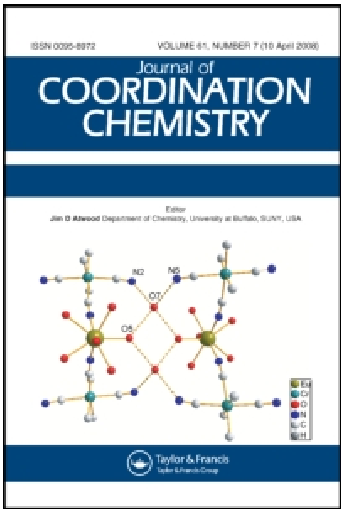

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Syntheses and crystal structures of nickel(II), copper(II), and zinc(II) complexes with a biphenyl-bridged bis(pyrrole-2-yl-methyleneamine) ligand

Guoqiang Yang[a]; Min Liu[a]; Xiuping Li[b]; Juan Li[b]; Jinshi Ma[b]
[a] Institute of Chemistry, Key Laboratory of Photochemistry, Chinese Academy of Sciences, Beijing, 100190, P. R. China [b] Department of Chemistry, Beijing National Laboratory for Molecular Sciences, Beijing, 100190, China

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Syntheses and crystal structures of nickel(II), copper(II), and zinc(II) complexes with a biphenyl-bridged bis(pyrrole-2-yl-methyleneamine) ligand

GUOQIANG YANG*†, MIN LIU†, XIUPING LI‡, JUAN LI‡
and JINSHI MA‡

†Key Laboratory of Photochemistry, Chinese Academy of Sciences,
Institute of Chemistry, No. 2, Bei Yi Jie, Zhong Guan Cun,
Beijing, 100190, P. R. China
‡Department of Chemistry, Beijing National Laboratory for
Molecular Sciences, Beijing, 100190, China

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The reactions of nickel(II), copper(II), and zinc(II) acetate salts with a potentially tetradentate biphenyl-bridged bis(pyrrole-2-yl-methyleneamine) ligand yielded three complexes with different coordination geometries. X-ray crystal structural analysis reveals that in the nickel(II) complex each nickel is five-coordinate, distorted trigonal bipyramid. In the copper(II) complex, each copper is four-coordinate, between square planar and tetrahedral. In the zinc(II) complex, each zinc is four-coordinate with a distorted tetrahedral geometry and the molar ratio of the zinc and ligand is 1 : 2.

Keywords: Nickel(II) complex; Copper(II) complex; Zinc(II) complex; Biphenyl-bridged Schiff-base ligand

1. Introduction

By changing the bridging spacers of Schiff bases and the metal centers, complexes with interesting structures can be constructed [1–7]. In our previous studies, we have employed bis(pyrrole) Schiff bases of flexible and rigid spacers to build diverse supramolecular complexes such as coordination polymers, helicates, molecular triangles and squares [8–10]. In this work, we use a biphenyl-bridged bis(pyrrole-2-yl-methyleneamine) to construct complexes with nickel(II), copper(II) and zinc(II). The conformation of the ligand and the coordination geometry of the metal centers are discussed in detail (scheme 1).

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*Corresponding author. Email: gqyang@iccas.ac.cn

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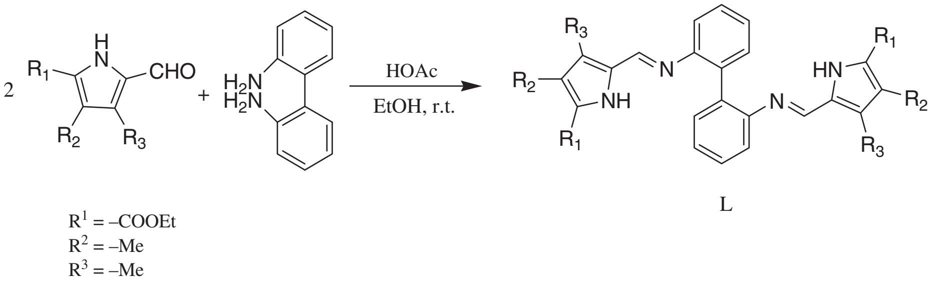

Scheme 1. Synthesis of ligand.

2. Experimental

2.1. *Materials and synthesis*

All reagents and solvents for synthesis were of analytical grade and used as received.

2.2. *Syntheses*

2.2.1. Ligand H_2L. Ethyl 5-formyl-3,4-dimethyl-1H-pyrrole-2-carboxylate [11] (0.781 g, 4 mmol) was dissolved in ethanol (10 mL) and a solution of 2,2′-diaminobiphenyl [12] (0.368 g, 2 mmol) in ethanol (10 mL) was added dropwise with stirring under argon at room temperature. After 5 min few drops of glacial acetic acid were added to the mixture. About 2 h later white precipitate appeared. Stirring was continued for another 4 h and the white precipitate was filtered, washed with cold ethanol, and dried under vacuum. Yield: 0.664 g (61.7%). ESI-MS: $m/z = 539.4$ $[M+H]^+$. ^1H NMR (400 MHz, $CDCl_3$, 25°C, TMS): $\delta = 9.23$ (br, 2H; NH), 7.97 (s, 2H; CH=N), 7.40 (p, $J = 7.6$ Hz, 4H, phenyl-H), 7.28 (t, $J = 7.2$ Hz, 2H, phenyl-H), 6.95 (d, $J = 7.6$ Hz, 2H, phenyl-H), 4.29 (q, $J = 7.2$ Hz, 4H, OCH_2CH_3), 2.22 (s, 6H, CH_3), 1.79 (s, 6H, CH_3), 1.33 (t, $J = 7.2$ Hz, 6H, OCH_2CH_3) ppm.

2.2.2. NiL(MeOH) (1). H_2L (0.215 g, 0.4 mmol) was dissolved in methanol (30 mL) under reflux; a solution of $Ni(OAc)_2 \cdot 4H_2O$ (0.010 g, 0.4 mmol) in methanol (10 mL) was added dropwise with stirring under argon. A few drops of triethylamine were added and the reaction continued overnight. The deep red-brown precipitate was filtered while hot, washed with hot methanol, and then dried under vacuum. Yield: 0.171 g (68.3%). Crystals suitable for X-ray analysis were obtained by slow evaporation from CH_2Cl_2 to MeOH (V/V : 2/1) solution. FT-IR (KBr, cm^{-1}): $\nu = 3438$(m), 3067(w), 2971(w), 2921(w), 1666(m), 1585(s), 1501(w), 1455(w), 1352(m), 1285(m), 1242(s), 1158(w), 1108(m), 1207(w), 934(w), 752(m). MALDI-TOF-MS: $m/z = 627$ $[M^+]$. Anal. Calcd for $C_{33}H_{36}NiN_4O_5$ (%): C, 63.2; H, 5.8; N, 8.9. Found (%): C, 63.0; H, 5.8; N, 8.9. UV-Vis: $\varepsilon = 2.95 \times 10^4 \text{ mol dm}^{-3} \text{ cm}^{-1}$ ($\lambda = 355$ nm).

2.2.3. CuL (2). A solution of H_2L (0.1080 g, 0.2 mmol) in dichloromethane (5 mL) was slowly added to a solution of $Cu(OAc)_2 \cdot H_2O$ (0.0399 mg, 0.2 mmol) in methanol

(5 mL) under argon. The resulting dark black solution was stirred for 5 min at room temperature and filtered. The filtrate had slowly evaporated in the dark at room temperature. After several weeks, black crystals suitable for X-ray analysis were collected by filtration. Yield: 0.044 g (36.7%). FT-IR (KBr, cm^{-1}): 3056(w), 2970(m), 2920(m), 1700(s), 1566(s), 1501(m), 1361(s), 1284(m), 1226(s), 1153(m), 1110(s), 937(m), 759(m). MALDI-TOF-MS: $m/z = 600$ [M^+]. Anal. Calcd for $C_{32}H_{33}CuN_4O_4$ (%): C, 63.9; H, 5.5; N, 9.3. Found (%): C, 63.9; H, 5.5; N, 9.4. UV-Vis: $\varepsilon = 4.49 \times 10^4\,mol\,dm^{-3}\,cm^{-1}$ ($\lambda = 377$ nm).

2.2.4. $Zn(HL)_2 \cdot 2MeOH$ (3). A solution of H_2L (0.108 g, 0.2 mmol) in 5 mL of dichloromethane was slowly added to 5 mL of methanol solution of $Zn(OAc)_2 \cdot 2H_2O$ (0.034 mg, 0.2 mmol). The resulting solution was stirred for 30 min at room temperature and filtered. The filtrate stood in the dark and was slowly evaporated at room temperature. After several weeks, yellow-green crystals suitable for X-ray analysis were collected by filtration. Yield: 0.066 g (54.5%). FT-IR: ν_{max} (cm^{-1}) 3452(w), 3267(w), 3056(w), 2975(w), 2922(w), 1698(s), 1572(s), 1246(s), 1110(m), 941(w), 760(m). ^1H NMR (400 MHz, $CDCl_3$, 25°C, TMS): $\delta = 9.26$(br), 8.10 (s, 2H; CH=N), 7.97 (s, 2H; CH=N), 7.41 (m, 12H, phenyl-H), 7.28 (m, 2H, phenyl-H), 6.99 (m, 2H, phenyl-H), 4.28 (q, 4H, OCH_2CH_3), 4.11 (m, 2H, OCH_2CH_3), 3.88 (m, 2H, OCH_2CH_3), 2.26 (s, 6H, CH_3), 2.22 (s, 6H, CH_3), 2.12 (s, 6H, CH_3), 1.79 (s, 6H, CH_3), 1.33 (t, 6H, OCH_2CH_3), 0.86 (t, 6H, OCH_2CH_3) ppm. MALDI-TOF-MS: $m/z = 1204$ [M^+]. Anal. Calcd for $C_{66}H_{74}ZnN_8O_{10}$ (%): C, 65.8; H, 6.2; N, 9.3. Found (%): C, 65.5; H, 6.1; N, 9.3. UV-Vis: $\varepsilon = 6.40 \times 10^4\,mol\,dm^{-3}\,cm^{-1}$ ($\lambda = 353$ nm).

2.3. *Physical measurements*

Elemental analyses were performed with a Carlo Erba-1106 elemental analyzer. FT-IR spectra were recorded on a BIO-RAD FT-165 IR spectrometer. ^1H NMR spectra were measured with a Bruker dmx 400 MHz spectrometer at room temperature in $CDCl_3$ with tetramethylsilane as an internal reference. The ESI-MS was carried out with a Bruker APEX II instrument. Molar absorptivities were obtained on a Hitachi UV-3010 absorption spectrograph.

2.4. *X-ray crystallography*

Accurate unit cell parameters were determined by a least-squares fit of 2θ values, measured for 200 strong reflections, and intensity data sets were measured on a Rigaku *R*-axis Rapid IP diffractometer with Mo-Kα radiation ($\lambda = 0.71073$ Å) at room temperature. The intensities were corrected for Lorentz and polarization effects, but no corrections for extinction were made. All structures were solved by direct methods. Non-hydrogen atoms were located in successive difference Fourier syntheses. The final refinement was performed by full-matrix least-squares methods with anisotropic thermal parameters for non-hydrogen atoms on *F*2. The hydrogens were added as ridings on the concerned atoms. In **2**, the ethyl of one ester group is disordered over two positions and refined isotropically with occupation factors of 0.75 and 0.25.

Table 1. Crystal data and structure refinement for **1**–**3**.

Complex	**1**	**2**[a]	**3**
Empirical formula	$C_{33}H_{36}N_4NiO_5$	$C_{32}H_{32}CuN_4O_4$	$C_{66}H_{74}N_8O_{10}Zn$
Formula weight	627.37	600.16	1204.70
Temperature (K)	293(2)	296(2)	294(2)
Crystal system	Triclinic	Triclinic	Monoclinic
Space group	$P\bar{1}$	$P\bar{1}$	$C2/c$
Unit cell dimensions (Å, °)			
a	9.4824(19)	8.9815(18)	24.136(4)
b	10.115(2)	13.294(3)	18.431(3)
c	17.845(4)	13.368(3)	14.080(2)
α	79.89(3)	77.69(3)	90
β	75.27(3)	87.60(3)	95.549(3)
γ	75.29(3)	72.08(3)	90
Volume (Å^3), Z	1589.8(6), 2	1483.2(5), 2	6234.1(17), 4
Calculated density (mg m^{-3})	1.311	1.344	1.284
Absorption coefficient (mm^{-1})	0.66	0.78	0.459
F(000)	660	626	658
Crystal size (mm^3)	0.47 × 0.40 × 0.31	0.79 × 0.55 × 0.21	0.24 × 0.20 × 0.16
Reflections collected	14,726	14,019	15,627
Unique reflections	7093	6729	5491
Independent reflection	[$R_{int} = 0.0619$]	[$R_{int} = 0.0362$]	[$R_{int} = 0.0577$]
Data/restraints/parameters	7093/0/390	6726/16/389	5491/37/395
Goodness-of-fit on F^2	1.015	1.027	1.030
Final R indices [$I > 2\sigma(I)$]	R_1[b] = 0.0730, wR_2[c] = 0.2045	R_1[b] = 0.0465, wR_2[c] = 0.1206	R_1[b] = 0.0618, wR_2[c] = 0.1638
R indices (all data)	R_1[b] = 0.0873, wR_2[c] = 0.2221	R_1[b] = 0.0623, wR_2[c] = 0.1282	R_1[b] = 0.1330, wR_2[c] = 0.2168
Largest difference peak and hole (e Å^{-3})	0.788, −0.726	0.380, −0.407	0.381, −0.585

[a] One disordered C_{31}–C_{32} group was refined with geometrical restraints applied; occupation factors were refined to 0.75 and 0.25, respectively.
[b] $R_1 = \Sigma||F_o|-|F_c||/\Sigma|F_o|$.
[c] $wR_2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^2)^2]^{1/2}$.

Crystallographic data for structure analyses are summarized in table 1 and the selected bond lengths and angles are listed in table 2.

3. Results and discussion

The Schiff-base H_2L was synthesized from the condensation of substituted 2-pyrrole-carboxaldehyde [11] and 2,2′-diaminobiphenyl [12] in molar ratio 2 : 1, with glacial acetic acid as the catalyst, which is less soluble in alcohol at room temperature. Complex **1** was obtained by the reaction of H_2L with nickel(II) acetate salt under refluxing with Et_3N, while **2** and **3** were formed at room temperature. Complexes **1**–**3** possess good solubility in THF, chloroform, dichloromethane, and DMF, but are less soluble in ethanol, methanol, acetonitrile, hexane, benzene, toluene, and water.

3.1. *Crystal structures of 1, 2, and 3*

The molecular structure of **1**, confirmed by single-crystal X-ray diffraction, is a mononuclear neutral complex. The nickels are not four-coordinate as usual but are

Table 2. Selected bond distances (Å) and angles (°) for **1**–**3**.

1			
Ni(1)–N(1)	2.024(3)	Ni(1)–O(5)	2.028(3)
Ni(1)–N(2)	2.034(3)	Ni(1)–N(4)	2.051(3)
Ni(1)–N(3)	2.106(3)		
N(1)–Ni(1)–O(5)	101.05(12)	N(1)–Ni(1)–N(2)	81.35(11)
O(5)–Ni(1)–N(2)	90.10(12)	N(1)–Ni(1)–N(4)	122.83(11)
O(5)–Ni(1)–N(4)	94.61(11)	N(2)–Ni(1)–N(4)	153.64(12)
N(1)–Ni(1)–N(3)	98.73(11)	O(5)–Ni(1)–N(3)	158.30(13)
N(2)–Ni(1)–N(3)	84.11(11)	N(4)–Ni(1)–N(3)	81.98(11)
2			
Cu(1)–N(2)	1.946(2)	Cu(1)–N(4)	1.963(2)
Cu(1)–N(1)	1.994(2)	Cu(1)–N(3)	2.003(2)
N(2)–Cu(1)–N(4)	113.73(9)	N(2)–Cu(1)–N(1)	83.91(9)
N(4)–Cu(1)–N(1)	149.16(9)	N(2)–Cu(1)–N(3)	149.16(9)
N(4)–Cu(1)–N(3)	82.72(8)	N(1)–Cu(1)–N(3)	94.47(8)
3			
Zn(1)–N(1)	1.935(4)	Zn(1)–N(1A)	1.935(4)
Zn(1)–N(2A)	2.111(4)	Zn(1)–N(2)	2.111(4)
N(1A)–Zn(1)–N(1)	130.4(2)	N(1)–Zn(1)–N(2A)	133.34(15)
N(1A)–Zn(1)–N(2A)	81.98(15)	N(1)–Zn(1)–N(2)	81.98(15)
N(1A)–Zn(1)–N(2)	133.34(15)	N(2A)–Zn(1)–N(2)	98.6(2)

Symmetry transformations used to generate equivalent atoms: A = $-x+1$, y, $-z+3/2$.

five-coordinate with methanol in the fifth position. The crystal structure of **1** reveals a distorted trigonal bipyramidal geometry [13–16].

The crystal structure of **1** is shown in figure 1. Each nickel is coordinated to four nitrogens of the ligand (two pyrroles and two imines) and an additional methanol resulting in five-coordination. The Ni(1)–N(2) distance is 2.034 Å while Ni(1)–N(3) is 2.106 Å, and the distances from Ni(1) to the pyrrole donors N(1) and N(4) are 2.024 and 2.051 Å, respectively. The Ni(1)–O(5) distance is 2.028(3) Å and the average Ni–N is 2.054 Å. The bond angles of N(1)–Ni(1)–N(2) (81.4°), N(1)–Cu(1)–N(4) (122.8°), and N(2)–Ni(1)–N(4) (153.6°) sum to 357.8° and the dihedral angles between the two Ni–N–N terminal planes is 101.3°, indicating that the two planes are nearly perpendicular. The O(5)–Ni(1)–N(3) (158.3(13)°) deviates from 180°. The coordination geometry of nickel may be described as distorted trigonal bipyramid with Ni(1), N(1), N(2), and N(4) forming the equatorial plane and O(5), N(3) occupying axial positions. The dihedral angle of the two phenyl rings of the biphenyl group is 53.8°, which is in the normal range for dihedral angles of phenyl rings in biphenyl compounds [17–22].

The ligand is tetradentate but the metal ions adopt trigonal bipyramid coordination, which is not very common. The trigonal bipyramid coordination geometries are usually formed with a tridentate ligand [13–16]. Schiff bases condensed from pyridylaldehyde or salicylaldehyde with unsubstituted [17, 18] or 6,6′-substituted 2,2′-diaminobiphenyl [19–22] and their metal complexes have been reported, but pentacoordinate complexes are not found with divalent metal ions in this system.

Figure 2 shows that **2** is mononuclear and each copper is intermediate between square planar and tetrahedral. The copper is coordinated to four nitrogens from two pyrrole-2-yl-methyleneamine units. The angles between the two planes, defined by Cu(1), N(1), N(2) and Cu(1), N(3), N(4), is 44.3°. The bond angles at copper N(1)–Cu(1)–N(2), N(3)–Cu(1)–N(4), N(1)–Cu(1)–N(3), N(2)–Cu(1)–N(4), N(4)–Cu(1)–N(1), and

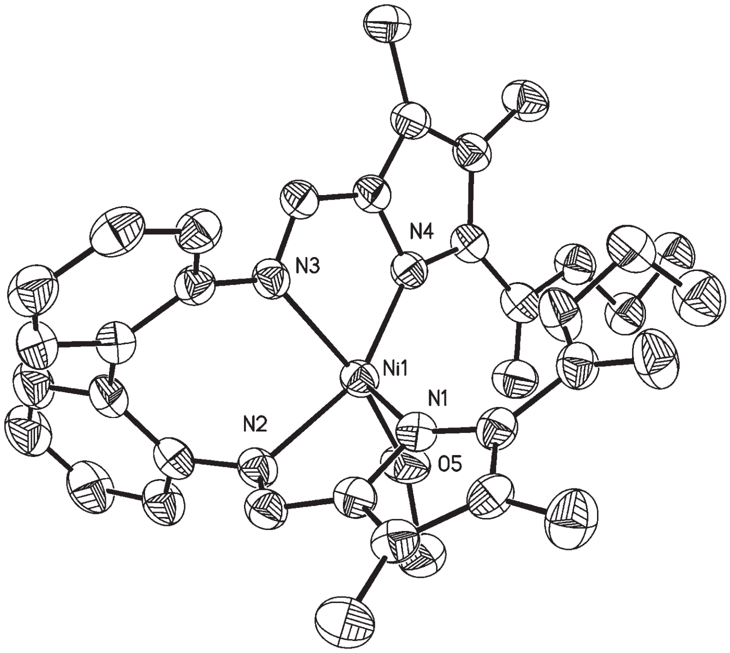

Figure 1. ORTEP drawing of **1** (30% thermal probability ellipsoids). Hydrogens are omitted for clarity.

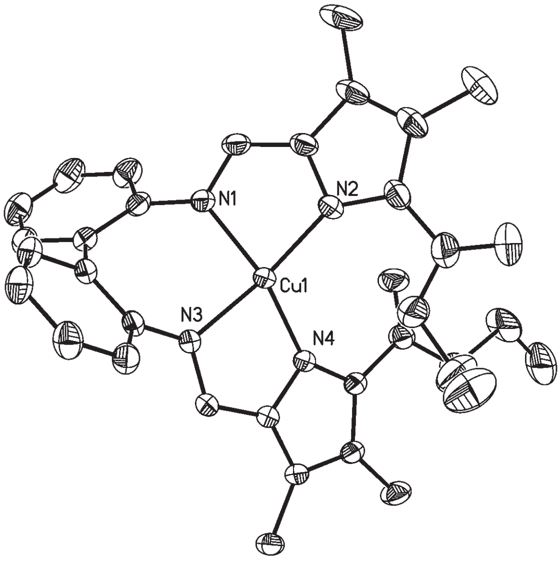

Figure 2. ORTEP of **2** (30% thermal probability ellipsoids). Hydrogens are omitted for clarity.

N(2)–Cu(1)–N(3) are 83.9, 82.7, 94.5, 113.7, 149.2, and 149.2°, which are not in accord with 109° for a tetrahedron. However, the four angles sum to 374.8° (close to 360°), indicating a more likely square-planar environment. The covalent Cu–N distance is in the range 1.9464–1.9631 Å and the dative Cu–N distance is between 1.9940 and

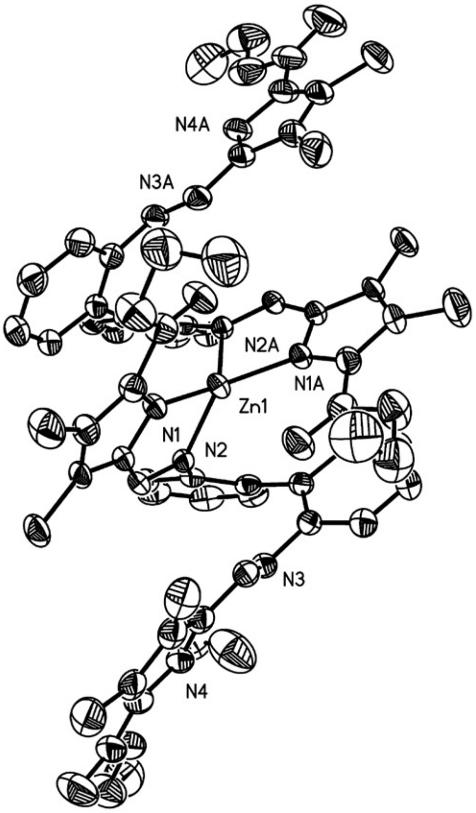

Figure 3. ORTEP of the asymmetric unit of **3** (30% thermal probability ellipsoids). Hydrogens and methanol are omitted for clarity.

2.0034 Å, in the normal range of Cu–N bonds [23–26]. The dihedral angle of the two phenyl rings of the biphenyl group is 53.1°.

The crystal structure of **3** is illustrated in figure 3 and the selected bond lengths and angles are summarized in table 2. Each molecule contains one zinc and two ligands in 1 : 2 molar ratio. The zincs are coordinated to four nitrogens belonging to two ligands, one nitrogen of pyrrole, and one nitrogen of imine for each ligand. Two methanols per molecule are present in the unit cell and the extra four nitrogens exhibit hydrogen bonding with them. The geometry of zinc is distorted tetrahedrally, although the two terminal bidentate fragments are not perpendicular to each other. The dihedral angle of the two terminal planes N(1)–Zn(1)–N(2) and N(1A)–Zn(1)–N(2A) is 67.5°. The bond angles of N(1)–Zn(1)–N(1A), N(1)–Zn(1)–N(2), N(2)–Zn(1)–N(2A), and N(1A)–Zn(1)–N(2A) are 130.4°(2), 81.98°(15), 98.6°(2), and 81.97°(15), respectively, also showing deviation from an ideal tetrahedron. The covalent Zn(1)–N(1) bond length is 1.935(4) Å, significantly shorter than the dative Zn(1)–N(2) distance of 2.111(4) Å. The dihedral angle of the two phenyl rings of the biphenyl fragment is 55.2°, similar to the reported values. Hydrogen bonding interactions exist between uncoordinated nitrogens and free methanol, indicated in figure 4. Hydrogen bond lengths of O(5)–N(3) and O(5)–N(4) are 2.829 and 2.796 Å, respectively, both in the normal range. The packing diagram is displayed in figure 5.

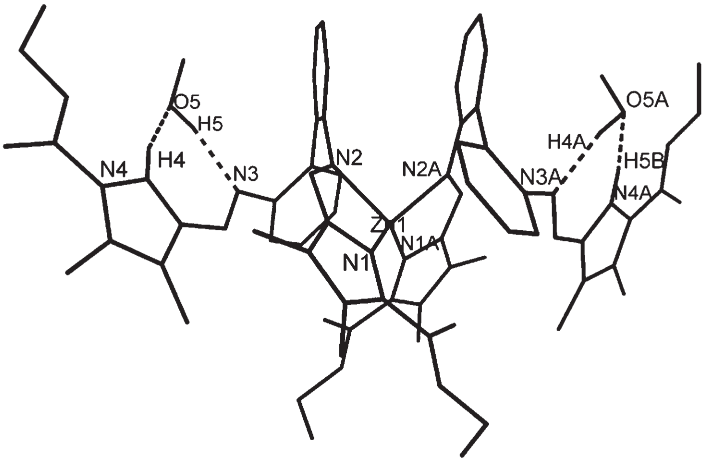

Figure 4. Hydrogen bonding interactions in **3**.

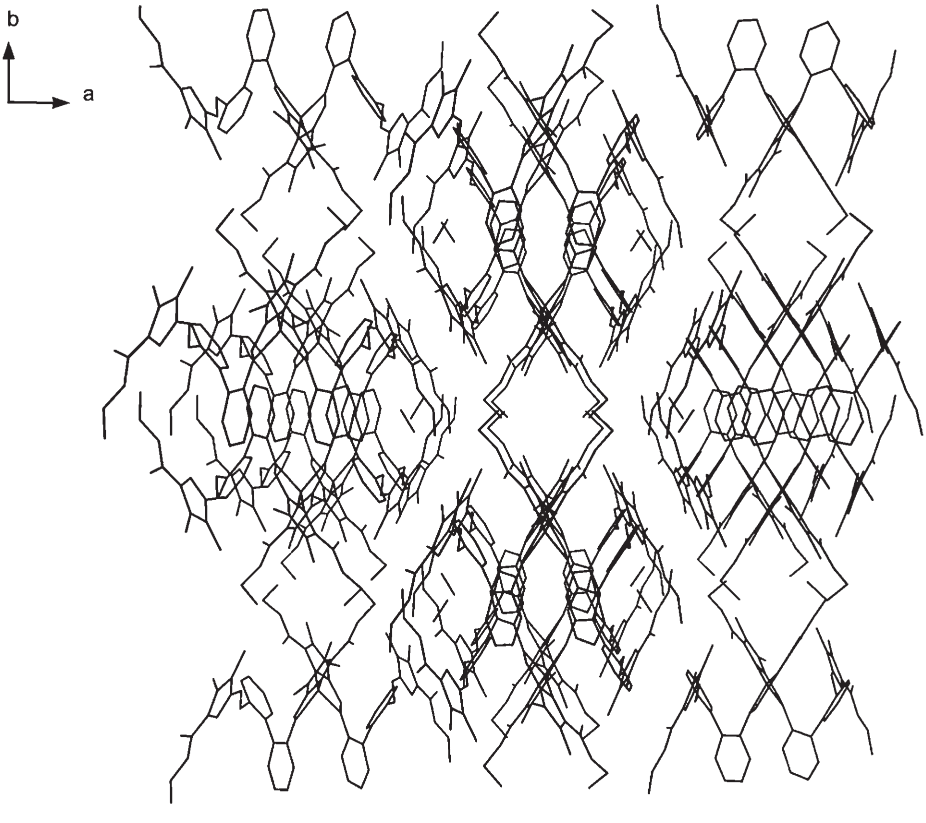

Figure 5. Packing diagram of **3** viewed along the *c*-axis.

The biphenyl-bridged Schiff base with different metal ions yielded three complexes with different structures. The conformation of the ligand is too distorted for metal ions to adopt simple tetrahedral or square planar geometry. The resulting structure is the corporate effect of the ligands and the metal ions. Similar ligands with aliphatic spacers assemble with metal ions; flexibility and length of spacers can result in forming helical complexes [27, 28]. Ligands with rigid spacers are difficult to construct such structures. Few articles have discussed this problem in detail. To make clear how spacers affect the resulting structures is a challenge, which attracts our further research.

4. Conclusion

We have synthesized three metal complexes based on a biphenyl-bridged pyrrole Schiff base. The nickel complex is five-coordinate trigonal bipyramidal, the copper complex is four-coordinate intermediate between square-planar and tetrahedral, and zinc complex are monomeric with two ligand molecules and a tetrahedral geometry.

Supplementary materials

Crystallographic data have been deposited with the Cambridge Crystallographic Data Centre as Supplementary Publications CCDC-647986, -647988 and -710961 for **1**, **2** and **3**, respectively. Copies of the data can be obtained via the Cambridge Crystallographic Data Centre [Email: deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk/deposit].

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