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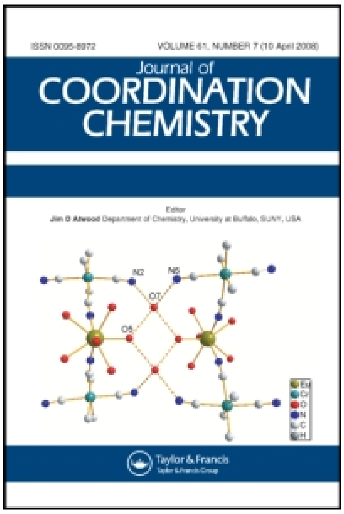

Journal of Coordination Chemistry
Publication details, including instructions for authors and subscription information:
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Spectroscopic and structural studies of 6,6'-*bis*(*N*-methylhydrazine)-2,2'-bipyridine and its mononuclear copper(II) complex

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First published on: 25 August 2010

To cite this Article Sengul, Abdurrahman , Yilmaz, Ismail , Sahin, Ertan and Karadayi, Nevzat(2010) 'Spectroscopic and structural studies of 6,6'-*bis*(*N*-methylhydrazine)-2,2'-bipyridine and its mononuclear copper(II) complex', Journal of Coordination Chemistry, 63: 19, 3453 — 3462, First published on: 25 August 2010 (iFirst)

To link to this Article: DOI: 10.1080/00958972.2010.512385

URL: http://dx.doi.org/10.1080/00958972.2010.512385

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Journal of Coordination Chemistry
Vol. 63, No. 19, 10 October 2010, 3453–3462

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Spectroscopic and structural studies of 6,6′-*bis*(*N*-methylhydrazine)-2,2′-bipyridine and its mononuclear copper(II) complex

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(*Received 12 February 2010; in final form 4 June 2010*)

The tetradentate ligand, 6,6′-*bis*(*N*-methylhydrazine)-2,2′-bipyridine (**L**) and its mononuclear copper(II) complex [Cu(**L**)](ClO_4)$_2$] (**1**) have been synthesized and characterized. The crystal structures of **L** and **1** have been determined by single-crystal X-ray diffraction. Both crystallize in the centrosymmetric monoclinic space group with crystallographic inversion symmetry. The ligand adopts a planar transoid configuration in the solid state. In **1**, the Cu(II) is six-coordinate octahedral, defined by N_4O_2 donors from ligand and two perchlorates. The molecular units are connected by intermolecular H-bonds between the hydrazino group of the one unit and coordinated perchlorate of the neighboring two units *via* N–H···O to furnish a 2-D network. Coordinated perchlorates also form an intramolecular H-bond with hydrazine influencing the crystal packing.

Keywords: Bipyridine; Copper complexes; N-donor ligands; Crystal structure; Hydrazine

1. Introduction

Simple hydrazine derivatives such as phenylhydrazine, *p*-nitrophenylhydrazine, and hydrazinopyridine (HP) attract attention in biology and coordination chemistry. In particular, interactions of HP with copper amine oxidases (CAOs) as well as its efficiency in ^{99}Tc-labeling of biologically active molecules have been studied [1–3]. Complexes of HP with Fe(II), Co(II), Ni(II), Cu(II), and Pd(II) have been previously reported [4–7]. Analogous compounds were also synthesized in an effort to obtain new complexes with decreased toxicity [8].

Hydrazine-substituted heterocyclic ligands are useful for preparation of a wide variety of pyrazolyl derivatives [9]. Free macrocycles incorporating bpy or phen have been synthesized by template condensation of 6,6′-*bis*(hydrazino)-2,2′-bipyridine or 2.9-*bis*(hydrazino)-1,10-phenanthroline with dicarbonyl ligands in the presence of

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Journal of Coordination Chemistry
ISSN 0095-8972 print/ISSN 1029-0389 online © 2010 Taylor & Francis
DOI: 10.1080/00958972.2010.512385

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Cr(III) [10] and also by cyclization of the corresponding hydrazino complexes of Ni(II) or Co(II) with aqueous acetone and other α-methyl ketones or aldehydes [11, 12]. These ligands also undergo Schiff-base condensation with a variety of aldehydes and ketones (i.e., 2-pyridinecarboxaldehyde [13, 14] or glyoxal [15] and butanedione [16]) to form multimodal ligands incorporating hydrazone linkers, which form binuclear double-stranded helical complexes with Co(II), Ni(II), Cu(II), Cu(I), Zn(II), and Ag(I) [13, 14, 16] and also a helical trinuclear silver(I) complex [15].

We have previously communicated the polymerization of 6,6′-*bis*(hydrazino)-2,2′-bipyridine with terephtaloylchloride and reported the synthesis of new polyhydrazides [17]. Herein, we present the syntheses, characterization, and single-crystal structures of 6,6′-*bis*(*N*-methylhydrazino)-2,2′-bipyridine (**L**) and its mononuclear copper(II) complex (**1**).

2. Experimental

2.1. *Materials and methods*

All chemicals were of reagent grade and used as received unless otherwise stated. DMSO and all other reagents were purchased from Sigma–Aldrich. *Caution!!!* Although no problems were encountered in this study, perchlorate salts containing organic ligands are potentially explosive. Only small amounts of the material should be prepared and handled with care.

2.2. *Physical measurements*

Elemental analysis data were obtained from a LECO CHNS analyzer. Nuclear magnetic resonance (NMR) measurements were performed using a Bruker DPX-400, 400 MHz high-performance digital Fourier transform-nuclear magnetic resonance (FT-NMR). Infrared (IR) spectra as KBr pellets were recorded using the Thermo Nicolet 6700 FT-IR spectrophotometer. Atmospheric pressure ionization–electrospray ionization (API–ESI) mass spectra were obtained from a liquid chromatography–mass spectrometry (LC–MS) Agilent 1100 MSD mass spectrometer. UV-Vis measurements were recorded using a Cary VinUV 100 Bio, Varian spectrophotometer.

2.3. *Crystallography*

For crystal structure determination, a single crystal of $C_{10}H_{12}O_4$ (**L**) was used for data collection on a four-circle Rigaku R-AXIS RAPID-S diffractometer (equipped with a 2-D area IP detector). The graphite-monochromated Mo-Kα radiation ($\lambda = 0.71073$ Å) and oscillation scans technique with $\Delta\omega = 5^\circ$ for one image were used for data collection. Lattice parameters were determined by least-squares methods on the basis of all reflections with $F^2 > 2\sigma(F^2)$. Integration of the intensities, correction for Lorentz, and polarization effects and cell refinement were performed using CrystalClear (Rigaku/MSC Inc., 2005) software [18]. Intensity data for **1** were collected using a Stoe-IPDS-2 diffractometer (Mo-Kα radiation, $\lambda = 0.71073$ Å) at 296 K [19].

Table 1. Crystallographic data for **L** and **1**.

	L	**1**
Empirical formula	$C_6H_8N_3$	$C_{12}H_{16}Cl_2CuN_6O_8$
Formula weight	122.15	506.75
Temperature (K)	293(2)	296(2)
Wavelength (Å)	0.71073	0.71073
Crystal system	Monoclinic	Monoclinic
Space group	$P2_1/c$	$P2_1/c$
Unit cell dimensions (Å, °)		
a	6.1490(5)	7.5856(4)
b	8.5440(5)	12.5548(5)
c	12.1410(5)	19.6823(12)
β	95.283(5)	95.343(5)
Volume (Å^3), Z	635.14(7), 4	1866.31(9), 4
Calculated density (g cm^{-3})	1.277	1.804
Absorption coefficient (mm^{-1})	0.084	1.513
θ range for data collection (°)	3.3–31.0	1.93–27.9
Limiting indices	$-8 \le h \le 8$; $-12 \le k \le 12$; $-17 \le l \le 17$	$-9 \le h \le 9$; $-16 \le k \le 16$; $-25 \le l \le 25$
Reflections collected	18,093	26,336
Data/parameters	1913/84	4441/281
Goodness-of-fit on F^2	1.138	1.039
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.088$	$R_1 = 0.041$
Largest difference peak and hole (e Å^{-3})	0.299 and 0.310	0.720 and 0.520

The structures were solved by direct methods using SHELXS-97 [20] and refined by a full-matrix least-squares procedure using SHELXL-97 [20]. H5a, H5b, H6a, and H6b of **1** were located in the difference Fourier map and refined independently with isotropic displacement parameters [N4–H6A = 1.00(4) Å, N4–H6B = 1.00(4) Å, N5–H5A = 0.86(4) Å, and N5–H5B = 0.81(3) Å]. The remaining hydrogens were positioned geometrically and refined using a riding model, fixing the aromatic C–H distances at 0.93 Å and methyl C–H distances at 0.96 Å [$U_{iso}(H) = 1.2U_{eq}$(C,N) and $U_{iso}(H) = 1.5U_{eq}$(methyl C)]. The final difference Fourier maps showed no peaks of chemical significance.

Details of crystal data, data collection, structure solution, and refinement for the structures of **L** and **1** are given in table 1.

2.4. *Synthesis of 6,6′-bis(N-methylhydrazine)-2,2′-bipyridine (L)*

Synthesis of **L** followed a similar procedure as that of 6,6′-*bis*(hydrazino)-2, 2′-bipyridine [17], except that *N*-methylhydrazine was used instead of hydrazine hydrate. The yellow solid was recrystallized from dichloromethane and afforded (87%) yellow needles, m.p. 158–160°C. API–ESI: m/z 245.1 $[MH]^+$. Anal. Calcd for $C_{12}H_{16}N_6$ (%): C, 59.0; H, 6.6; and N, 34.4. Found: C, 59.1; H, 6.6; and N, 34.4. UV-Vis (MeOH): λ_{max}, nm: 228, 262, and 346. δ_H (400 MHz, $CDCl_3$, 25°C, TMS): 7.74(t, $J = 16$ Hz, 2H, bpy-3,3′), 7.67(t, $J = 7$ Hz, 2H, bpy-4,4′), 7.11(d, $J = 8$ Hz, 2H, bpy-5,5′), 3.44(s, 6H, $2 \times CH_3$), and 2.7(s, 4H, $2 \times NH_2$). IR (KBr, ν, cm^{-1}): 3300, 3197, 2923, 2843, 1639, 1577, 1557, 1477, 1441, 1402, 1364, 1290, 1227, 1130, 1066, 1022, 974, 914, 790, and 632.

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2.5. *Synthesis of the copper(II) complex (1)*

The **L** (0.02 g, 0.08 mmol) was dissolved in dry MeOH (20 mL) and added to a methanol solution of $Cu(ClO_4)_2 \cdot 6H_2O$ (0.03 g, 0.08 mmol). The mixture was stirred vigorously at 50–60°C for 24 h. The obtained green solution was concentrated under reduced pressure and filtered off. The filtrate was left as such at room temperature to yield needle-shaped single crystals.

[Cu(L)(ClO_4)$_2$] **1**: Yield 70%. Anal. Calcd for $C_{12}H_{16}N_6Cl_2O_8Cu$ (%): C, 28.5; H, 3.2; and N, 16.6. Found: C, 28.6; H, 3.3; and N, 16.6. API–ESI: *m*/*z* 406.0 [Cu(**L**)](ClO_4)]$^+$. UV-Vis (MeOH): λ_{max}, nm: 220, 262, and 372. IR (KBr, ν, cm^{-1}): 3453, 3250, 3070, 2990, 2923, 1627, 1570, 1500, 1210, 1144, 1139, 1087, 1014, 786, 628, and 459.

3. Results and discussion

3.1. *Synthesis of L*

α-Diimines such as bpy and phen have been extensively studied as simple bidentate ligands to form complexes of high redox stability with many of the transition metals [21], but relatively little is known about hydrazine-substituted bpy or phen as a quadridentate ligand. Complexes of the 6,6′-*bis*(hydrazino)-2,2′-bipyridine and **L** have been reported previously for Cr(III) [10], Ni(II) [11], and Co(II) [12]. However, there have been no previous structural reports on the ligands and their complexes. Synthesis and spectroscopic characterization of the ligand have been previously published [17]. The synthesis of **L** *via* reaction of 6,6′-dichloro-2,2′-bipyridine with *N*-methyl hydrazine was carried out under dry N_2. Recrystallization of the yellow product from dichloromethane gave yellow needles suitable for X-ray analysis. Microanalysis and mass results confirm the structure. The ^{1}H-NMR shows C_2-symmetry with hydrazine protons as a singlet at 2.7 ppm and *N*-methyl as a singlet at 3.44 ppm. Integration is consistent with the structure. In the IR spectrum, strong absorptions at 3300 and 3198 cm^{-1} are characteristic for stretching vibrations of NH_2; absorptions at 2924 and 2843 cm^{-1} are attributed to C–H vibrations of the *N*-methyl groups. Strong absorptions at 1640–1478 cm^{-1} are C=N and C=C combination vibrations. The stretching mode of the N–N bond is at 974 cm^{-1} [22].

3.2. *Crystal structure of L*

The molecular structure of **L** with atomic labeling is shown in figure 1, while selected bond distances and angles are given in table 2. The molecule crystallizes in the space group *P2$_1$/c*, lying on a center of symmetry, with nitrogens of pyridine rings *trans* to each other around the central bond [C(5)–N(1)–C(1)–C(1a) = 179.1(2)°], similar to that of 6,6′-dimethyl-2,2′-bipyridine [23]. Weak intermolecular interactions between hydrazine hydrogens and pyridine nitrogens of adjacent molecules stabilizes the molecule in the solid state (table 3). Bond lengths and angles are as expected [23–26]. The structure arises from a combination of packing effects, steric effects, and very

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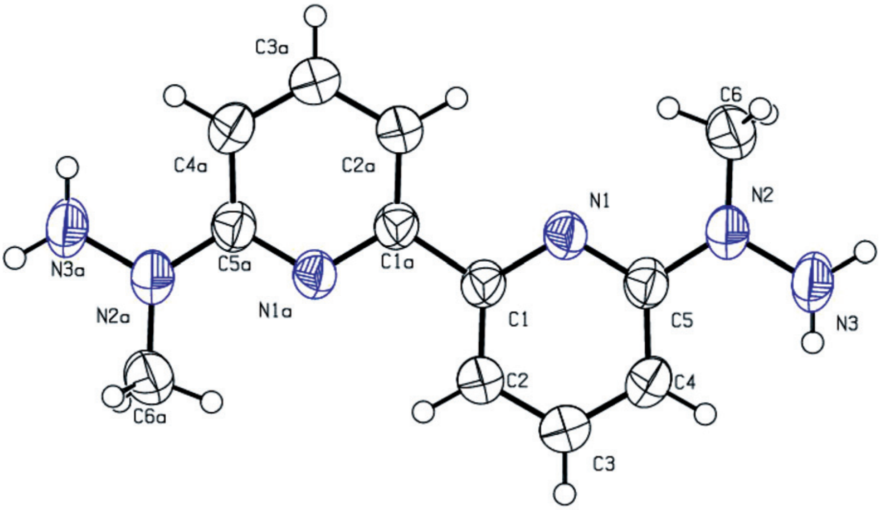

Figure 1. Structure of **L** with atomic labeling.

Table 2. Selected bond distances and angles for **L** and **1**.

L^{a}		
N(1)–C(1) = 1.354(3)	N(1)–C(1)–C(1a)$^{\mathrm{i}}$ = 115.8(2)	
N(2)–C(6) = 1.427(4)	N(1)–C(5)–N(2) = 117.5(3)	
N(1)–C(5) = 1.338(3)		
C(5)–N(2) = 1.364(4)		
C(1)–C(1a)$^{\mathrm{i}}$ = 1.486(3)		
C(5)–C(4) = 1.405(4)		
N(3)–N(2) = 1.404(3)		
1		
N(1)–C(5) = 1.350(3)	N(6)–N(5)–Cu(1) = 108.9(2)	C(11)–N(3)–N(4)–Cu(1) = 173.6(2)
N(1)–C(1) = 1.332(3)	C(1)–N(1)–Cu(1) = 118.1(2)	Cu(1)–N(5)–N(6)–C(12) = 174.1(2)
C(1)–N(6) = 1.352(4)	N(2)–Cu(1)–N(4) = 80.6(2)	C(5)–N(1)–Cu(1)–N(5) = 175.5(2)
N(6)–N(5) = 1.429(3)	N(1)–Cu(1)–N(5) = 80.5(2)	N(3)–N(4)–Cu(1)–N(1) = 15.5(4)
C(5)–C(6) = 1.492(4)	N(3)–N(4)–Cu(1) = 109.1(2)	N(3)–N(4)–Cu(1)–N(5) = −170.2(2)
N(6)–C(12) = 1.438(2)	C(10)–N(3)–N(4) = 117.5(2)	N(3)–N(4)–Cu(1)–N(2) = 6.45(2)
Cu(1)–N(1) = 1.907(2)	O(3)–Cu(1)–O(5) = 162.6(4)	N(1)–C(5)–C(6)–N(2) = 2.2(4)
Cu(1)–N(5) = 2.035(2)	O(3)–Cu(1)–N(1) = 97.1(6)	C(10)–N(2)–C(6)–C(5) = 177.9(3)
Cu(1)–N(2) = 1.920(2)	O(3)–Cu(1)–N(2) = 92.0(3)	
Cu(1)–N(4) = 2.026(2)	N(2)–Cu(1)–N(5) = 160.0(3)	
Cu(1)–O(3) = 2.572(2)		
Cu(1)–O(5) = 2.535(1)		
Cl(1)–O(3) = 1.425(5)		
Cl(2)–O(5) = 1.428(7)		
C(5)–C(6) = 1.492(4)		
Cl(1)–O(2) = 1.420(2)		

^{a}Symmetry code: $^{\mathrm{i}}-x, -y+1, -z$.

weak hydrogen contacts; the molecule contains C–H$\cdots$N and N–H$\cdots$N interactions throughout the structure (figure 2). Additionally, N(3)–H$\cdots$*Cg* (pi-ring)$^{\mathrm{i}}$ (symmetry $^{\mathrm{i}}1-x, -y, -z$) interactions (H$\cdots$*Cg* = 2.61 Å, N$\cdots$Cg = 3.507(4) Å) contribute to the stabilization.

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Table 3. Hydrogen bond parameters for **L** and **1**.

D–H···A	D–H (Å)	H···A (Å)	D···A (Å)	∠D–H···A
L[a]				
N(3)–H···N(1)[i]	1.00	2.23	3.180(4)	158
C(6)–H···N(1)	0.96	2.34	2.783(4)	107
1[b]				
N(5)–H···O(8)	0.86	2.53	3.114(1)	126
N(5)–H···O(2)[i]	0.86	2.40	3.145(3)	145
N(5)–H···O(7)[ii]	0.81	2.35	3.119(5)	158
N(4)–H···O(4)	1.00	2.08	2.913(3)	139
N(4)–H···O(7)[iii]	1.00	2.42	3.060(4)	121
N(4)–H···O(2)[iv]	1.00	2.13	3.041(7)	150
N(4)–H···O(8)[iii]	1.00	2.58	3.182(3)	118
C(2)–H···O(4)[v]	0.93	2.47	3.293(4)	148
C(11)–H···O(1)[iv]	0.96	2.53	3.270(1)	134
C(12)–H···O(6)[ii]	0.96	2.59	3.188(1)	121

[a] Symmetry code: [i] $1-x$, $-1/2+y$, $1/2-z$.

[b] Symmetry codes: [i] $-x$, $1-y$, $-z$; [ii] $-1+x$, y, z; [iii] $1-x$, $1-y$, $-z$; [iv] $1+x$, y, z; and [v] x, $3/2-y$, $-1/2+z$.

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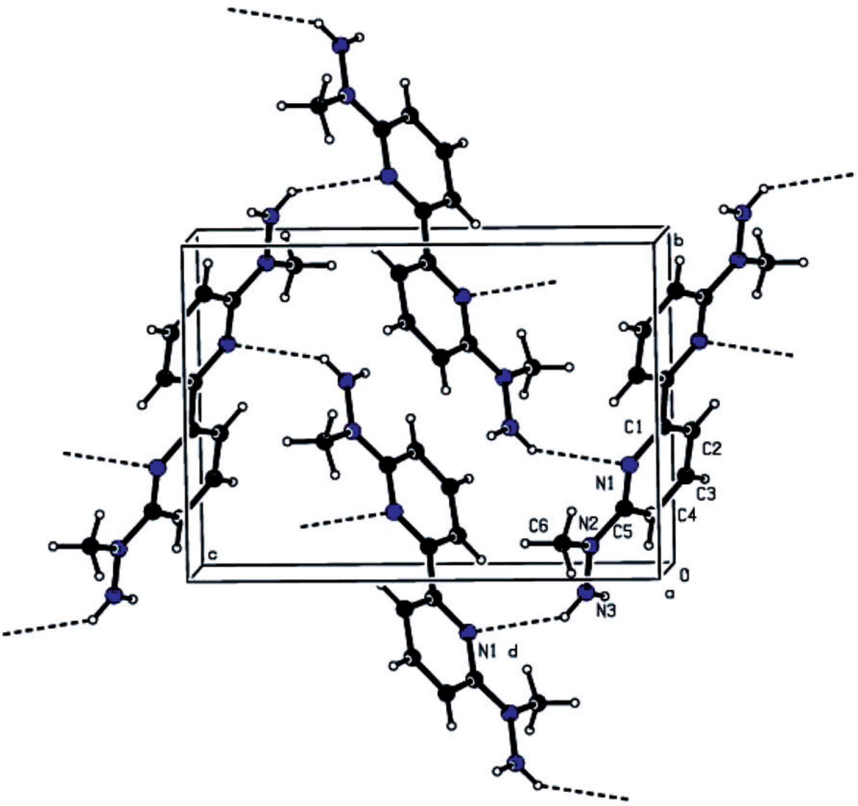

Figure 2. Packing diagram of **L** showing hydrogen bonds (dashed lines).

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3.3. *Synthesis and characterization of copper(II) complex (1)*

The copper(II) complex, **1**, was synthesized by the reaction of **L** with the $Cu(ClO_4)_2 \cdot 6H_2O$ in methanol, where the concentrated solution yielded X-ray quality green crystals. In the ESI–MS spectrum, the molecular ion peak corresponding to **1** was observed at 406.0 and supports the formulation [Cu(**L**)$(ClO_4)_2$], as well as stability in solution. The microanalysis gave satisfactory results and agrees well with the structure.

In the IR spectrum of **1**, bands in the mid-IR range are mostly due to the stretching vibrations of the hydrazine moieties and "in-plane" vibrations of the pyridine rings. Generally, after coordination, the position of some vibrations would change. Thus, in the case of **1**, the absorption band at 3453 cm^{-1} is very strong and broad due to hydrogen bonding of NH_2. The second stretching vibration which appears as a shoulder at 3250 cm^{-1} can be attributed to an overtone of the N–H bending vibration at 1627 cm^{-1} [27]. The aromatic C–H vibration appears at 3070 cm^{-1}. Bands at 2990 and 2923 cm^{-1} are assigned to $\nu_{(CH)}$ vibration of methyl groups [22]. The C=N vibration shifts to a lower frequency at 1627 cm^{-1} with lower intensity. The C=C vibrations appear at 1577 and 1557 cm^{-1} in the free ligand. The vibration bands in the free ligand at 914, 790, and 632 cm^{-1} have been replaced by new bands at 1014, 786, and 628 cm^{-1}. In addition, the presence of a new band at 459 cm^{-1}, which is absent in the free ligand, may indicate the coordination of hydrazine to the metal ion. The splitting of the perchlorate band at 1088 and 1114 cm^{-1} is an indication of the presence of a coordinated perchlorate [28].

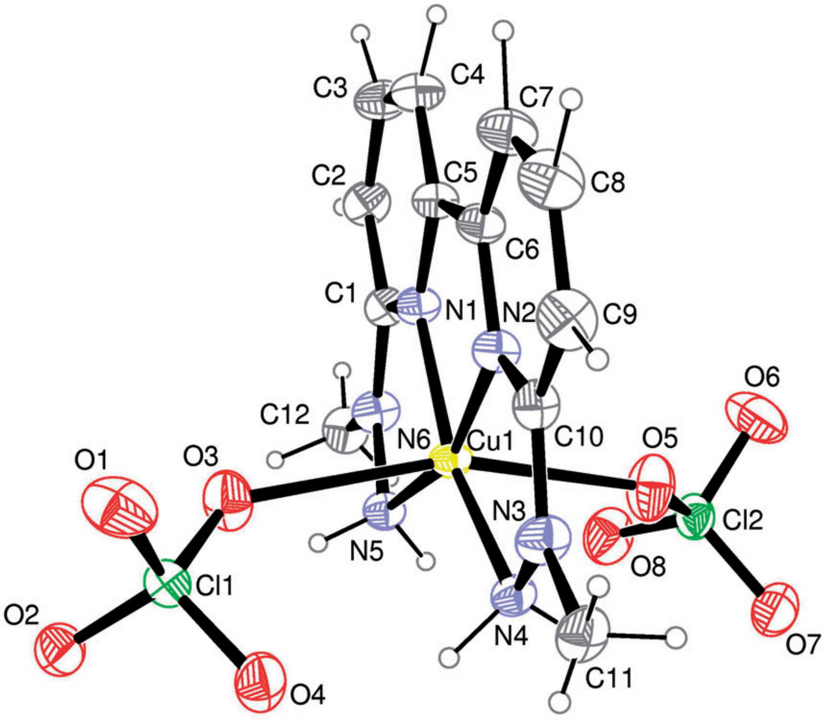

Figure 3. Structure of **1** with atomic labeling.

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3.4. *Crystal structure of 1*

An Oak Ridge Thermal Ellipsoid Plot Program (ORTEP) view of **1** together with the atom numbering scheme is shown in figure 3. The coordination polyhedron around copper is best described as a distorted octahedron. The four nitrogens [two pyridine

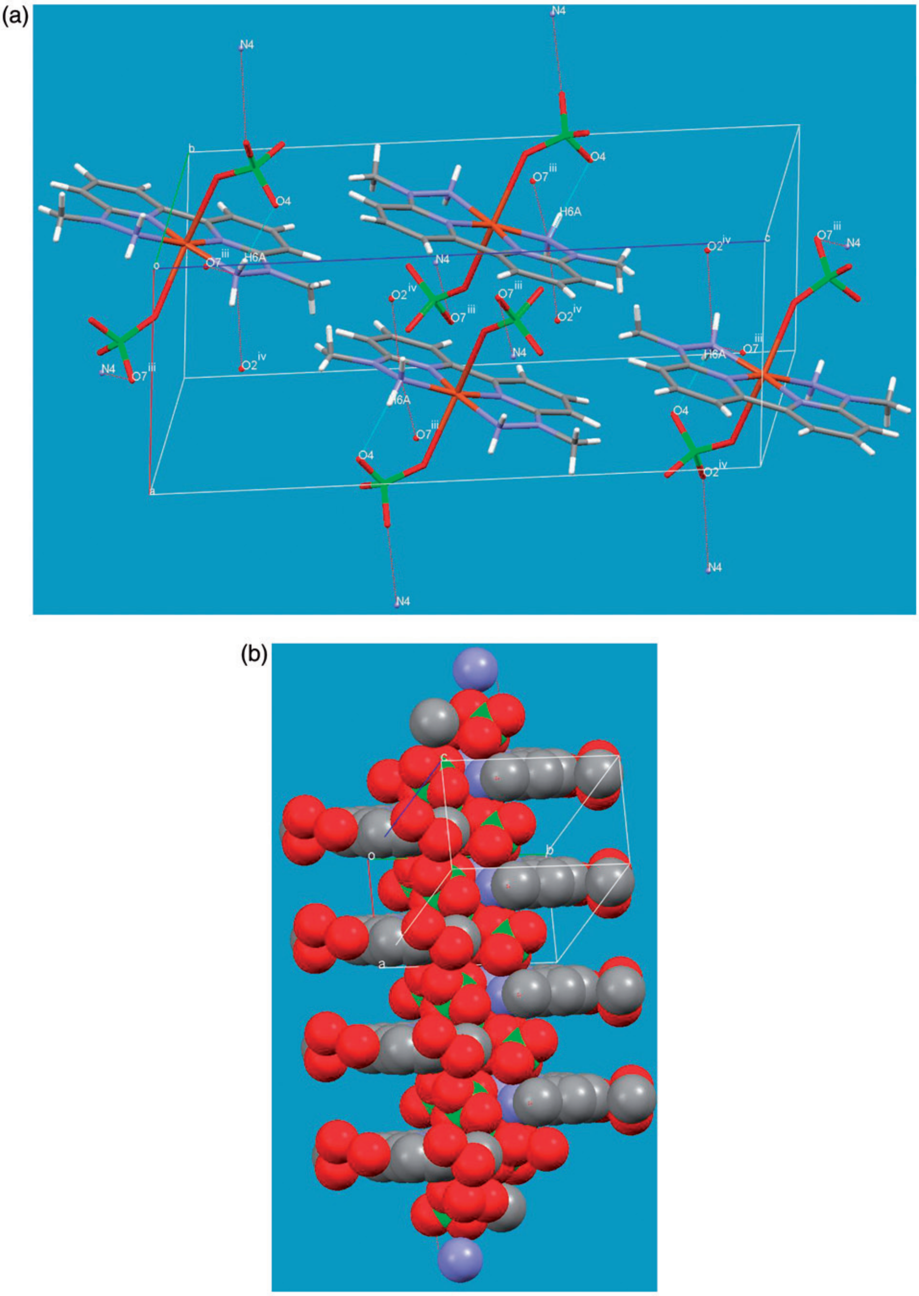

Figure 4. (a) Unit cell of **1** showing hydrogen bonds. (b) Packing of **1**.

N(1), N(2) and two hydrazine N(5), N(4)] of the tetradentate ligand define the equatorial plane. Two oxygens, O(3) and O(5), of two perchlorates are coordinated *trans* at distances of 2.573(2) and 2.535(2) Å (table 2). Long bonds in the axial direction, compared with those in the equatorial plane [Cu(1)–N(1) = 1.907(2) Å, Cu(1)–N(2) = 1.920(2) Å, Cu(1)–N(4) = 2.026(2) Å, Cu(1)–N(5) = 2.035(2) Å], are usually observed in octahedral copper complexes [29, 30], from Jahn–Teller effect.

The values of dihedral Cu(1)–N(1)–C(1)–N(6)–N(5) and C(5)–N(1)–C(1)–C(2) angles demonstrate that the chelates and pyridine rings are almost coplanar, confirmed by a dihedral angle of 2.42(1)° between their planes.

The copper(II)–hydrazine nitrogen distances are longer than those of pyridine and are close to those observed for palladium(II) complexes with HP [21] and the copper(II) complex with 2-hydrazino-1,10-phenanthroline [31] and 2-HP [6], showing that the aliphatic amine groups coordinate much more weakly than their counterparts. The formation of the five-membered chelate rings causes the N(1)–Cu(1)–N(5) and N(2)–Cu(1)–N(4) angles to be only 80.5(2)° and 80.6(2)°, respectively. The O(3)–Cu(1)–O(5) angle of 162.6(4)° deviates from linearity, presumably due to hydrogen bonding with hydrazine hydrogens (table 3).

The complex units form hydrogen bonds with NH_2 as donors and perchlorates as acceptors. This type of bonding, shown in figure 4, stabilizes the crystal structure. The structure contains classical N–H ··· O hydrogen bonds and non-classical C–H ··· O hydrogen bonds, as shown in table 3. Each coordinated perchlorate takes part in intramolecular hydrogen bonding with the hydrazine NH_2 group [N(4)–H ··· O(4) 2.08(3) Å and 139.0(1)° and N(5)–H(5A) ··· O(8) 2.53(3) Å and 126.0(1)°]. The 1-D chains are further connected by N–H ··· O hydrogen bonds to generate a 2-D network as depicted in figure 4 (data of hydrogen bonds are summarized in table 3). There are five N–H ··· O intermolecular hydrogen bonds between hydrazine NH_2 and coordinated perchlorate. The coordinated perchlorates also participate in formation of three C–H ··· O nonclassical hydrogen bonds with the ligands from three adjacent units (table 3).

4. Conclusion

This study demonstrates the synthesis of a tetradentate ligand, *bis*(*N*-methylhydrazino)-2,2′-bipyridine, and its coordination behavior toward copper(II). The copper(II) is coordinated by the ligand in a distorted octahedral fashion *via* N_{bpy}, $N_{hydrazine}$, and $O_{perchlorate}$. The ligand and the copper(II) complex have been characterized by various physicochemical investigations and also by single-crystal X-ray analysis. The crystal structure of the ligand shows that ligand adopts a *trans* geometry with C_2-symmetry, whereas it has a *cis* conformation in **1**.

Supplementary material

Crystallographic data for the structural analyses of **L** and **1** have been deposited with the Cambridge Crystallographic Data Centre, CCDC nos 708847 and 708848, respectively. Copies of this information may be obtained free of charge from the

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Acknowledgments

The authors are grateful to the Turkish Scientific and Technical Research Council [TBAG-2450 (104T060)] and also Zonguldak Karaelmas University for providing grant 2007/3-13-02-01.

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