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Two new copper(II) complexes with the shortest (N–N) diazine based rigid ligand: Example of unusual tridentate coordination mode

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Abstract

Two new five coordinated Cu(II) complexes, $Cu(L)Cl_2,CH_3OH$ (**1**) and $Cu(L)Br_2$ (**2**) derived from the flexidentate ligand (L), 2-pyridinealdazine, have been synthesised and characterised by spectroscopic and electrochemical studies. Single crystal structures of the complexes were determined. Crystal structures of both the complexes contain monomeric entities of five coordinated copper(II) ions where the Schiff base ligand, 2-pyridinealdazine, acts in a tridentate fashion. The central part of the ligand in complex **2** is disordered over two positions: N8—N9 make up the major position and N8A—N9A make up the minor position.

Keywords: Copper(II) monomers; Flexidentate Schiff base ligand; Crystal structure; Spectral studies; Electrochemical properties

1. Introduction

Many copper(II) complexes are generally biologically active due to their chelating ability and positive redox potential. In particular, some copper(II) complexes posses a wide range of biological activity as antivirals, fungicides, pesticides and even tracers to medicine [1–4] depending on the ligand binding sites.

In order to design species presenting specific structural and functional features, it is of great importance to establish the rules by which control of the self-assembly process can be achieved through chemical programming of suitable components and assembling algorithms [5]. Dinucleating diazine ligands containing a single N–N bond have received considerable attention over the years beginning with the early work of Butch on pyridinealdazine (PAA) [6,7]. Compared with the diazine moiety in heterocyclic ring systems the N_2 diazine linkages in open chain systems with N–N single bonds are much more flexible [8]. Previous studies [9–13] showed that such open chain diazine ligands present several possible mono- and dinucleating coordination modes due to the flexibility of the ligand around the N–N single bond. In combination with other donors such ligand can form several types of, e.g. mono- [13–16], di- [17–23], tri- [24] and tetranuclear copper(II) complexes [25,26].

The simplest bis-pyridylimine ligand system is 2-pyridinealdazine (L) in which two pyridylimine binding units are linked directly (no spacer unit) through the imine nitrogen atoms [27].

The different possible arrangements of L, 2-pyridinealdazine are shown in Scheme 1.

The binuclear and helical composition are consistent with the coordination abilities of *E–E* or *Z–E*, respectively [13]. Stratton and Busch described the coordination of L to octahedral transition metal ions to form complexes with the formula $[M_2(L)_3]$ (M = Co, Fe, Ni and L = 2-pyridylmethylketazine) and recognised that the species must consist of three stands wrapped around two metals in a spiral fashion [6,7,9,28,29]. The authors proposed the term “flexidentate” to describe the coordination behaviour of this type of ligand.

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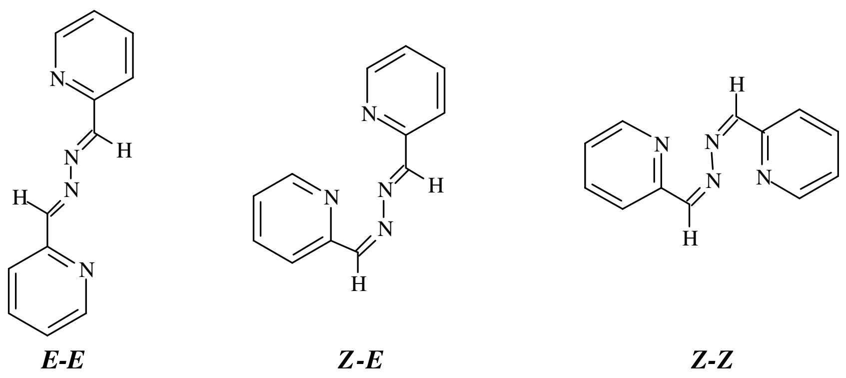

Scheme 1.

No attempt has been made to isolate the various stereoisomers of bis-2-pyridylmethylaldazine or ketazine before reacting the ligand with the copper(II) ions. Previously, other workers have unsuccessfully attempted to separate the stereoisomers and come to the conclusion that the stable form of the ligand is *E–E*. The *Z–E* isomer can be generated by a template rearrangement reaction of solvated ligand and solvated metal ion. *Z–E* isomer is only stable when coordinated to a metal centre [9,10,28,29]. Another recent study by Dong showed a novel triangular Ni(II) complex of the 2-pyridinealdazine ligand, where the imine based ligand behaves as a rigid one [30].

In some cases, the spiral or helical complexes are shown to undergo exchange reactions to form mononuclear complexes $[ML_2]^{2+}$ (L = 2-pyridinealdazine) in which the ligand twists to coordinate as a tridentate one but this phenomenon is still not been well explored [27].

In this paper, we report the synthesis, spectral characterisation, electrochemical studies and crystal structure of two new unusual five coordinated Cu(II) complexes, $CuLCl_2,CH_3OH$(**1**) and $CuLBr_2$(**2**) derived from the flexidentate ligand L, 2-pyridinealdazine which coordinates to the metal ion in a rare *Z–E* fashion.

2. Experimental

2.1. Materials

Reagent grade pyridine 2-carboxaldehyde, hydrazine hydrate, copper(II) chloride and copper(II) bromide (Fluka) were obtained commercially. All the solvents used were of reagent grade.

2.2. Measurements

Infrared spectra (4000–350 cm^{-1}) and far-IR spectra (400–50 cm^{-1}) of complexes **1** and **2** were recorded on a Perkin-Elmer FT-IR and FTIR Bomen DA 8.3 spectrophotometers, respectively, and electronic spectra on a Perkin-Elmer Lambda-40 spectrophotometer using acetonitrile as solvent. Elemental analyses were carried out on a Perkin-Elmer 2400-II instrument. Electrochemical studies were performed on a CH 600A cyclic voltameter instrument using acetonitrile as solvent and tetrabutyl ammonium perchlorate as supporting electrolyte at a scan rate of 50 mV s^{-1}. Magnetic susceptibility data were collected on a model 155 PAR vibrating sample magnetometer fitted with a waker scientific 175 FBAL magnet using $Co[Hg(SCN)_4]$ as the standard.

2.3. Preparation of the ligand and complexes

2.3.1. Synthesis of the Schiff base ligand (L)

A methanolic solution (20 ml) of pyridine-2-carboxaldehyde (1.90 ml, 20 mmol) was mixed with a methanolic solution (10 ml) of hydrazine hydrate (0.50 ml, 10 mmol). The resulting mixture was stirred at room temperature for half an hour when yellow crystalline solid separated out. The yellow solid Schiff base ligand was filtered and recrystallised from a methanol-acetonitrile (1:1) mixture.

2.3.2. Synthesis of $CuLCl_2\cdot CH_3OH$ (1)

To a methanolic solution of copper(II) chloride (0.170 g, 1 mmol), methanolic solution of the Schiff base ligand (0.210 g, 1 mmol) was added with constant stirring. The resulting deep green solution was stirred for 10 min at room temperature. After 3 days green crystals suitable for X-ray diffraction were collected and rest were filtered and air-dried.

Yield: 65 %,

Anal. Found: C, 41.36; H, 3.70; N, 14.79; Cu, 16.85; calc. for $C_{13}H_{14}Cl_2Cu N_4O$, C, 41.41; H, 3.72; N, 14.86; Cu, 16.89 %.

2.3.3. Synthesis of $CuLBr_2$ (2)

Complex **2** was synthesised following the same procedure as in complex **1**, except using copper(II) bromide instead of copper(II) chloride.

Yield: 60 %,

Anal. Found: C, 33.20; H, 2.27; N, 12.88; Cu, 14.62; calc. for $C_{12}H_{10}Br_2CuN_4$, C, 33.21; H, 2.31; N, 12.91; Cu, 14.68 %.

2.4. X-ray data collection and structure refinement

Single crystal data and details of the structure determinations for complexes **1** and **2** are presented in Table 1.

Table 1
Crystallographic data for complex **1** and **2**

	1	**2**
Emperical formula	$C_{13}H_{14}Cl_2CuN_4O$	$C_{12}H_{10}Br_2CuN_4$
Formula weight	376.72	433.60
Crystal system	Monoclinic	Monoclinic
Space group	P2(1)/c	P2(1)/n
a (Å)	7.2349(2)	8.1278(6)
b (Å)	13.8531(3)	14.879(1)
c (Å)	14.7814(4)	11.2331(7)
β (°)	90.967(1)	92.698(3)
Cell volume (Å^3)	1481.27(7)	1356.95(16)
Z	4	4
D_c (Mg m^{-3})	1.689	2.122
$F(000)$	764	836
μ (mm^{-1})	1.838	7.488
T (K)	123(2)	123(2)
λ (Å)	0.71073	0.71073
Theta range (°)	3.25–27.89	3.17–27.86
Index ranges	$-9 <= h <= 7$	$-10 <= h <= 10$
	$-18 <= k <= 18$	$-19 \leqslant k \leqslant 19$
	$-19 <= l <= 19$	$-14 \leqslant l \leqslant 14$
Max. and min. transmission	0.8745 and 0.6086	0.7059 and 0.1913
Total no. of reflections measured	18501	13700
Total no. of unique reflections	3494 [R(int) = 0.0790]	3198 [R(int) = 0.1125]
No. of observed reflections ($I > 2\sigma(I)$)	2614	2225
Data/restraints/parameters	3494/0/181	3198/2/190
Final R indices (obs. data)	$R_1 = 0.0712$, $wR_2 = 0.1692$	$R_1 = 0.0781$, $wR_2 = 0.2051$
Final R indices (all data)	$R_1 = 0.0985$, $wR_2 = 0.1861$	$R_1 = 0.1163$, $wR_2 = 0.2346$

Data were collected on a green-blue dichroic block ($0.08 \times 0.15 \times 0.30$ mm) for **1** and on a dark red plate ($0.05 \times 0.33 \times 0.33$ mm) for **2** at 123 K on a Nonius KappaCCD diffractometer with graphite monochromated Mo-$K\alpha$ radiation ($\lambda = 0.71073$ Å), using ϕ and ω rotations with 1° frames. The images were processed with the HKL suite of programs [31]. Solutions were obtained using SHELXS-97 [32] followed by successive difference Fourier methods, and structures were refined against F^2 using SHELXL-97 [32a]. Face-indexed absorption corrections were applied [32b]. All non-hydrogen atoms were refined anisotropically. For complex **1** all hydrogen atoms were assigned to calculated positions except the methanol hydroxy proton, which was neither found nor assigned.

For complex **2** the central part of the ligand is disordered over two positions: N8-N9 make up the major position (60%), with N8A-N9A making up the minor position (40%). All non-hydrogen atoms were refined anisotropically. Aromatic hydrogen atoms were assigned to calculated positions and not refined. The hydrogen atoms bonded to C7 and C10 were refined with the restraint that they were 0.84(2) Å from the corresponding carbons, while hydrogens were not assigned to the disordered nitrogen atoms.

3. Results and discussion

3.1. Infrared spectra

Stratton and Busch [33–36] have relied on the C–N stretching region in the IR spectrum to determine whether the ligand PAA is tridentate or tetradentate, forming mononuclear complexes with five membered and six membered chelate rings. The tridentate chelation of the ligand is accompanied by a distinct spliting of the pyridine ring breathing vibration (which appear in our case at 1021 and 1010 cm^{-1}) and of the C–N stretching mode of the azine link (at 1628 and 1584 cm^{-1}). This also proves that all the pyridine rings are coordinating. The absorption bands at 440 and 340 cm^{-1} can be assigned to the ν(Cu–N) modes where N corresponds to the azomethylene and the pyridine nitrogens, respectively [16].

For complex **1**, the absorption band appearing above 3000 cm^{-1} corresponds to the ν(OH) stretching vibration [37]. Above 1635 cm^{-1}, two strong ν(C=N) bands are observed for both the complexes **1** and **2**.

Far infrared bands at 276 and 253 cm^{-1} for complexes **1** and **2**, respectively, suggest the presence of terminal Cu—Cl or Cu—Br bonds, respectively.

3.2. Electronic spectra

The electronic spectra of complexes **1** and **2** in acetonitrile show bands in the region 210–345, 380–470 and 720 nm corresponding to intraligand charge transfer and d–d transitions, respectively.

The bands at 215 and 222 nm (for **1** and **2**) are attributable to the ring π–π* transitions [38], while absorptions in the 275–295 and 325–340 nm ranges for both **1** and **2** are assignable to the $n \rightarrow \pi*$ transitions of the pyridine ring [39]. The broad bands at 719 and 722 nm for **1** and **2**, respectively, are consistent with the effective five coordinated geometries observed at the copper(II) centres [40].

3.3. Electrochemical studies

Electrochemical behaviours of both the complexes are very similar. Fig. 1 represents the cyclic voltammogram of the complex **1**. Both voltammograms show only one reductive response on the negative side of SCE (at −1.24 V for complex **1**, −1.27 V for complex **2**), both of which are irreversible in nature. These are due to the Cu(II) to Cu(I) redox couple.

Similarly, irreversible oxidation responses are also observed on the positive side of SCE for both **1** and **2** (at 1.51 and 1.54 V, respectively) and these are tentatively assigned to oxidation of the coordinated ligands.

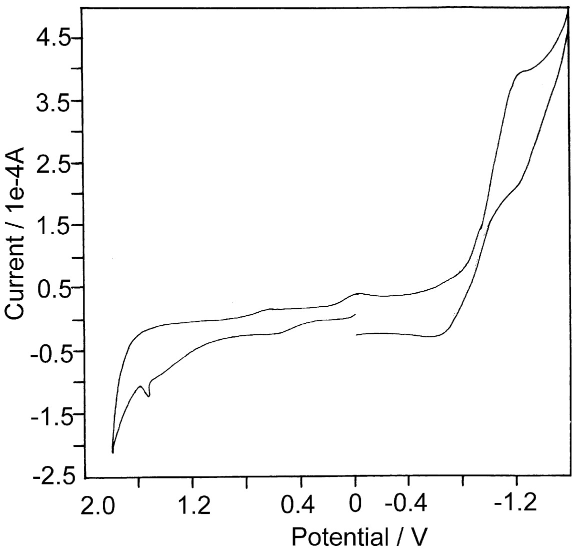

Fig. 1. Cyclic voltammogram of complex **1**.

3.4. Magnetic studies

The room temperature magnetic moments for the compounds **1** and **2** are 1.90 and 1.99 BM, respectively. The results are consistent with the presence of one unpaired electron in the Cu(II) (d^9) system.

3.5. Description of crystal structures

3.5.1. Crystal structure of complex *1*

The crystal structure of complex **1** contains monomeric entities of five coordinated copper(II) ions and one methanol molecule present in the lattice. A view of the molecular structure with atom numbering scheme is shown in Fig. 2 and selected bond lengths and angles are given in Table 2. The copper(II) ion is surrounded by three nitrogens [the N1 (2.039(5) Å), N8b (1.997(6) Å) and N16 (2.018(5) Å) atoms of the $C_5H_4NC{=}N{-}N{=}CNH_4C_5$ ligand], a terminal chloride ion Cl1 (2.5913(15) Å) in the basal plane, and another terminal chloride ion Cl2 (2.2662(13) Å) in an apical position.

Distortions for the coordination polyhedron from extreme square–pyramidal (SPY) and trigonal–bipyramidal (TBPY) topologies have been analysed by using the method of Addison et al. [41]. The value of $\tau = 0.122$ clearly indicates that the environment of the copper(II) ion is close to SPY topology. Deviation from ideal SPY geometry is indicated by the difference in *cisoid* (89.9(3)° to 94.55(14)°) and *transoid* angles (161.4(2)° to 168.8(2)°). Values are comparable to those found in similar systems [13,36]. The N1, N8b, N16 and Cl2 atoms are displaced 0.117, 0.110, 0.072 and 0.078 Å, respectively, from the mean basal plane and copper(II) ion sits 0.229 Å above the plane.

The methanol molecules hydrogen bond in centrosymmetrically related pairs (O1···O1 = 2.69(3) Å). Each methanol molecule also makes a weak interaction to adjoining Cl1 ligands (O1···Cl1 = 3.15(2) Å). The complex pack in

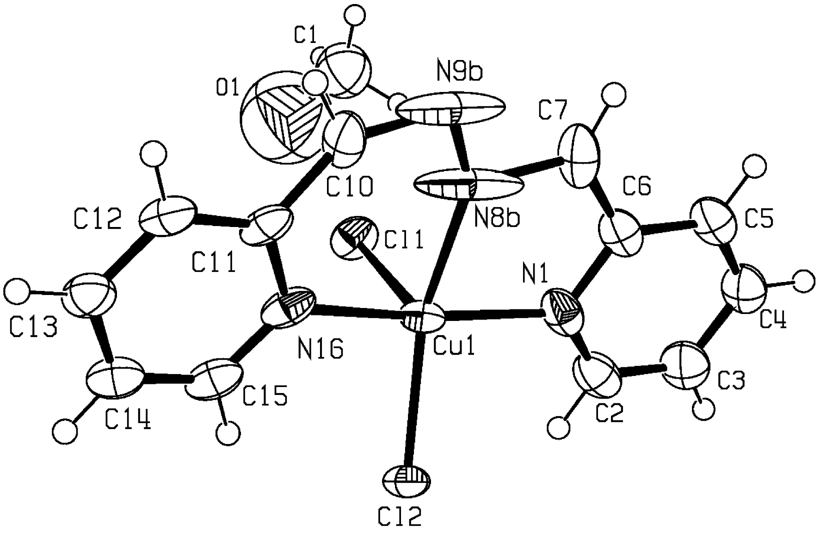

Fig. 2. A view of complex **1** with atom numbering scheme.

Table 2
Selected bond lengths (Å) and angles (°) for complex **1**

Cu(1)—N(8b)	1.997(6)	Cu(1)—N(16)	2.018(5)
Cu(1)—N(1)	2.039(5)	Cu(1)—Cl(2)	2.2662(13)
Cu(1)—Cl(1)	2.5913(15)		
N(8b)—Cu(1)—N(16)	89.9(3)	N(8b)—Cu(1)—N(1)	80.7(4)
N(16)—Cu(1)—N(1)	168.8(2)	N(8b)—Cu(1)—Cl(2)	161.4(2)
N(16)—Cu(1)—Cl(2)	94.55(14)	N(1)—Cu(1)—Cl(2)	92.50(16)
N(8b)—Cu(1)—Cl(1)	94.92(16)	N(16)—Cu(1)—Cl(1)	94.98(13)
N(1)—Cu(1)—Cl(1)	91.97(14)	Cl(2)—Cu(1)—Cl(1)	102.59(5)
N(1)—C(6)—C(7)—N(8b)	−0.4(7)	C(6)—C(7)—N(8b)—N(9b)	179.5(5)
C(7)—N(8b)—N(9b)—C(10)	−172.6(5)	N(8b)—N(9b)—C(10)—C(11)	0(1)
N(9b)—C(10)—C(11)—N(16)	−3(1)		

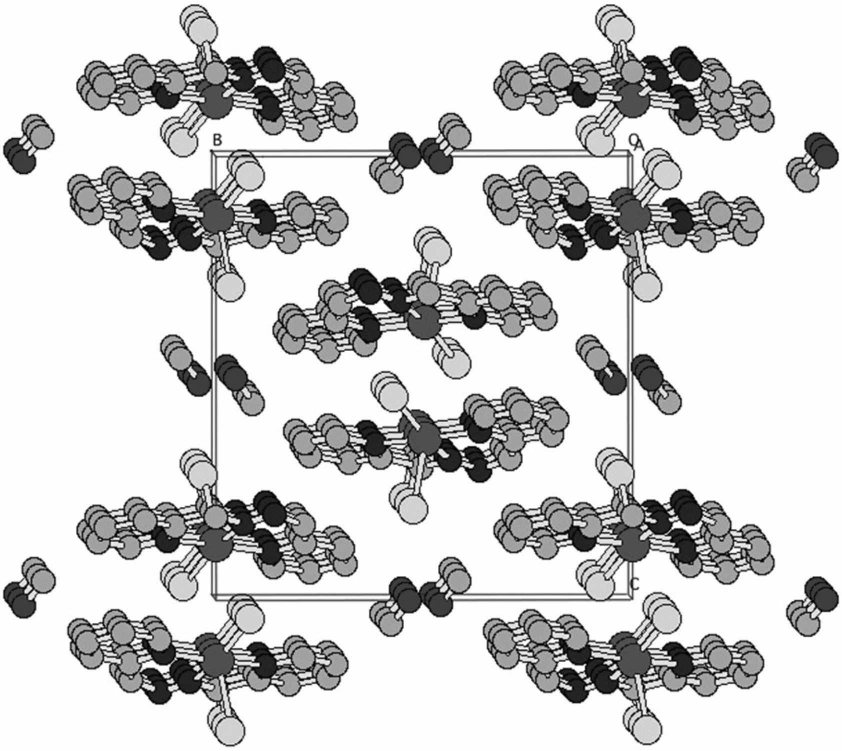

Fig. 3. Packing of complex **1** view down *a* axis (hydrogens omitted for clarity).

centrosymmetrically related pairs ‘back-to-back’ (Fig. 3), such that the Cl2 atoms of each complex make a long interaction to the adjoining copper atom (Cu1···Cl2 = 3.304 Å) *trans* to the axial Cl1 atoms. These pairs then stack in such a way that the pyridyl rings overlap in a π–π stacking fashion (closest C···C contact is 3.40 Å).

As seen in Table 2, the bond distances of the ligand in complex **1** are similar to those found in the free ligand or in similar systems [42–44] where the organic portion is stabilised as a neutral species.

3.5.2. Crystal structure of complex 2

The crystal structure of [$CuBr_2(C_5H_4NC{=}N{-}N{=}CNH_4C_5)$], complex **2** contains monomeric entity of five coordinated copper(II) ion. A view of the molecular structure is shown in Fig. 4. The selected bond lengths and angles are given in Table 3.

The ligand is disordered over two positions in the crystal structure: one position involving C7=N8—N9=C10, and the other position containing C7=N8a—N9a=C10. The copper(II) ion is surrounded by two bromide ions, Br1 (2.5103(15) Å) and Br2 (2.5074(15) Å), and one nitrogen from the ligand, N8a (2.08(2) Å) or N9 (2.016(14) Å), which are in the equatorial positions. The apical sites are occupied by two nitrogen atoms, N1(1.992(8) Å), and N16 (2.005(7) Å), of the $C_5H_4NC{=}N{-}N{=}CNH_4C_5$ ligand forming a trigonal–bipyramidal geometry.

The distortions for the coordination polyhedron from the extreme square–pyramidal (SPY) and trigonal–bipyramidal (TBPY) topologies have been analysed by using the method of Addison et al. [41]. The value of $\tau = 1.02$ clearly indicates that the environment of the copper(II) ion is close to TBP topology. The deviation from ideal TBP geometry is indicated by the difference in *cisoid* (106.59(5)° to

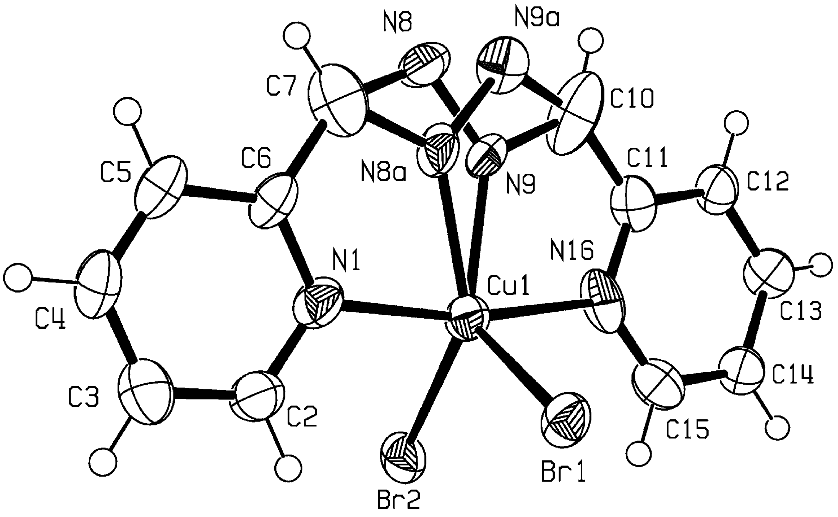

Fig. 4. A view of complex **2** with atom numbering scheme.

Table 3
Selected bond lengths (Å) and angles (°) for complex **2**

Cu(1)—N(1)	1.992(8)	Cu(1)—N(16)	2.005(7)
Cu(1)—N(9)	2.016(14)	Cu(1)—N(8A)	2.08(2)
Cu(1)—Br(2)	2.5074(15)	Cu(1)—Br(1)	2.5103(15)
N(1)—Cu(1)—N(16)	168.1(4)	N(1)—Cu(1)—N(9)	93.4(5)
N(16)—Cu(1)—N(9)	75.2(5)	N(1)—Cu(1)—N(8A)	72.8(7)
N(16)—Cu(1)—N(8A)	96.1(7)	N(9)—Cu(1)—N(8A)	21.0(6)
N(1)—Cu(1)—Br(2)	93.3(2)	N(16)—Cu(1)—Br(2)	92.0(3)
N(9)—Cu(1)—Br(2)	133.1(4)	N(8A)—Cu(1)—Br(2)	136.0(6)
N(1)—Cu(1)—Br(1)	95.4(2)	N(16)—Cu(1)—Br(1)	93.3(3)
N(9)—Cu(1)—Br(1)	118.9(4)	N(8A)—Cu(1)—Br(1)	115.9(6)
Br(2)—Cu(1)—Br(1)	106.59(5)		
N(1)—C(6)—C(7)—N(8)	6(3)	N(1)—C(6)—C(7)—N(8a)	0(1)
C(6)—C(7)—N(8)—N(9)	−3(3)	C(6)—C(7)—N(8)—N(9a)	−178(1)
C(7)—N(8)—N(9)—C(10)	180(1)	C(7)—N(8a)—N(9a)—C(10)	172(2)
N(8)—N(9)—C(10)—C(11)	179(1)	N(8a)—N(9a)—C(10)—C(11)	−11(4)
N(9)—C(10)—C(11)—N(16)	−4(1)	N(9a)—C(10)—C(11)—N(16)	6(4)

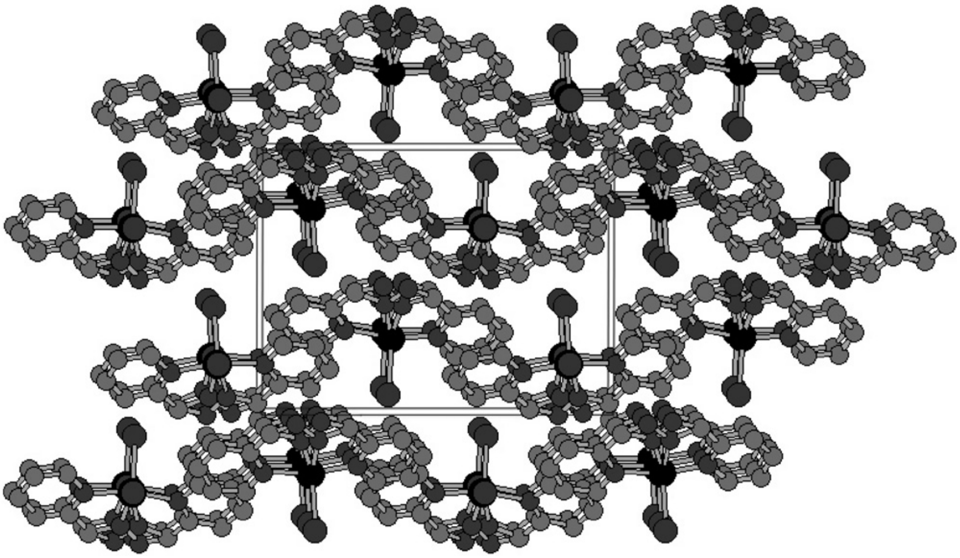

Fig. 5. Packing diagram of complex 2 view down *a* axis (*b* axis horizontal; *c* axis vertical, hydrogen atoms omitted for clarity).

136.0(6)° considering both the positions involving C7H=N8—N9=C10H and C7H=N8a—N9a=C10H) and *transoid* angles (168.1(4)°). The values are nearly comparable to those found in similar systems [13,16].

In the packing (Fig. 5) there are close π–π interactions between pyridyl rings (closest C···C = 3.33 Å), which give a zigzag arrangement of molecules which propagate along the *b* axis. Interactions between these chains are less pronounced, with the closest non-hydrogen contact being 3.51 Å.

As seen in Table 3, the bond distances of the complex **2** are very much similar, within experimental error, to those found in the free ligand or in similar systems [42–44] where the organic portion is stabilised as a neutral species.

4. Supplementary material

Crystallographic data for structural analysis have been deposited with the Cambridge Crystallographic Data Centre, CCDC Nos. 295773 for **1** and 295774 for **2**. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (fax: +44-1223-336033; deposit@ccdc.cam.ac.uk or http://www/.ccdc.cam.ac.uk).

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.molstruc.2006.04.040.

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