Intermolecular interaction and magnetic coupling mechanism of a mixed-valence Cu(II)Cu(I) tetranuclear complex with iodide bridge

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A tetranuclear Cu^{I}Cu^{II} mixed oxidation state complex, [Cu$^{II}_2$(μ-I)$_2$Cu$^{I}_2$(μ-I)$_2$(phenP)$_2$I$_2$] (phenPE: 2-(1H-pyrazol-1-yl)-1,10-phenanthroline), has been prepared and its crystal structure is determined by X-ray crystallography. In the complex, Cu^{II} is a distorted square pyramid and Cu^{I} is a distorted trigonal planar coordination environment; Cu^{II} and Cu^{I} are bridged by iodide. It is rare to form a Cu^{II}-iodide bond and for Cu^{II} and Cu^{I} to be bridged by iodide. In the crystal, there is a slipped π–π stacking between adjacent Cu^{II} complexes, which resulted in the formation of the 1-D chain along the *c* axis. The fitting for the variable-temperature magnetic susceptibility data gave magnetic coupling constant $2J = -1.16\,\mathrm{cm}^{-1}$ and it may be ascribed to the intermolecular π–π magnetic coupling pathway.

Keywords: Crystal structure; Magnetic coupling; Intermolecular interaction; Copper complex

1. Introduction

Molecular magnetism has attracted attention and major advances have been made in both their description and application as new molecular-based materials [1, 2]. Major research has concentrated on systems where coupling spin-carriers (or paramagnetic centers) are linked by bridging ligands and the magnetic interactions are through bond exchange [3–12]. In magnetic coupling, intermolecular interaction plays an important role. For example, some authors attributed strong ferromagnetic order to π–π stacking interaction [13] and other authors found that the π–π stacking interaction led to a

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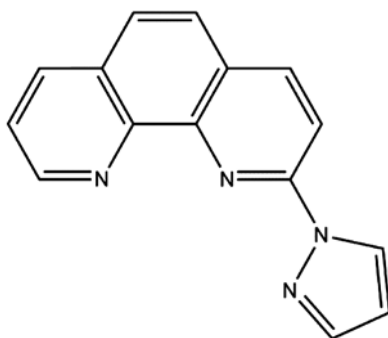
Scheme 1.   Structure of 2-(1H-pyrazol-1-yl)-1,10-phenanthroline.

strong antiferromagnetic interaction between spin-carriers [14–16]. Strong antiferromagnetic coupling between Cu^{II} ions through O–H$\cdots$O hydrogen bonding has also been reported [17]. The intermolecular force should be a factor that dominates magnetic coupling properties. However, not much work has been published on π–π stacking systems [13–16], hydrogen bond systems [18–25], and X–H$\cdots\pi$ interaction systems [21] in comparison to bond exchange systems. Most of these papers only deal with radicals [18, 25] or complexes [16, 26–29] with radicals as the ligands. Factors that dominate magnetic coupling properties have not been clearly understood. Thus, it is important to design and synthesize complexes with intermolecular interactions and to study the factors that dominate the magnetic coupling mechanism, an area to which our attention has been given [30–36].

2-(1H-pyrazol-1-yl)-1,10-phenanthroline, as shown in scheme 1, possesses strong chelated coordination group and a larger conjugation plane, which may be useful to form complexes with both strong π–π stacking and relevant magnetic coupling pathways. These intermolecular forces could be effective magnetic coupling pathways leading to interesting magnetic interactions. So far, no complex with 2-(1H-pyrazol-1-yl)-1,10-phenanthroline as the ligand has been published. Cu^{I}-iodide bonds and Cu^{I} ions bridged by an iodide are common [37–41], whereas complexes with Cu^{II}-iodide bond [42–46] are limited due to the reducing nature of iodide. Therefore, it is rare for mixed valence Cu^{II} and Cu^{I} to be bridged by iodide. Herein, we report a mixed-valence $Cu^{I}Cu^{II}$ tetranuclear complex with an iodide bridge and 2-(1H-pyrazol-1-yl)-1,10-phenanthroline as a terminal ligand and its magnetic coupling mechanism.

2. Experimental

2.1. *Materials*

Synthesis of 2-(1H-pyrazol-1-yl)-1,10-phenanthroline: 2-chloro-1,10-phenanthroline (2.13 g, 10 mM), pyrazole (0.70 g, 10 mM), and potassium carbonate (1.50 g, 0.011 mM) were dissolved in 20 mL of DMF. The mixture was refluxed for 24 h, then another batch of pyrazole (0.34 g, 5.0 mM) and potassium carbonate (1.00 g, 7.2 mM) was added into the DMF solution and the reaction continued to reflux for another 48 h. After that, a fraction of DMF was distilled out by vacuum and 40 mL of ice water with crushed ice was added into

the solution. The raw product then appeared and was separated by filtration. Then, the product was washed with ice water until the filtrate was neutral and the final product was obtained by vacuum drying. ^{1}H NMR (300 MHz, CDCl$_3$, 25 °C): $\delta = 9.19$ (s, 2H), 8.40 (brs, 2H), 8.28 (s, 1H), 7.82 (brs, 3H), 7.66 (s, 1H), 6.56 (s, 1H). HRMS (ESI): m/z calcd for C$_{15}$H$_{10}$N$_4$ + H$^+$: 247.0978 [M+H$^+$]; found: 247.0982. All other chemicals are analytical grade and used without purification.

2.2. Preparation of complex

About 8 mL methanol solution containing Cu(ClO$_4$)$_2$·6H$_2$O (0.0749 g, 0.20 mM) was added into 10 mL acetonitrile solution of 2-(1H-pyrazol-1-yl)-1,10-phenanthroline (0.0506 g, 0.21 mM) and then 5 mL methanol solution of NaI (0.0380 g, 0.20 mM) was added; the mixed solution was stirred for a few minutes. Red single crystals were obtained after the filtrate was allowed to slowly evaporate at room temperature for one week. IR (cm^{-1}): 1612(m), 1591(m), 1532(s), 1503(s), 1393(m), 1356(s), 955(m), 856(s), 775(s). Elemental Anal. Calcd for C$_{15}$H$_{10}$Cu$_2$I$_3$N$_4$: (Fw 754.05) C, 23.89; H, 1.34; N, 7.43; Cu, 16.85%. Found: C, 23.61; H, 1.02; N, 7.78; Cu, 17.22%.

2.3. Physical measurements

Infrared spectra were recorded with a Bruker Tensor 27 infrared spectrometer from 4000 to 500 cm^1 using KBr disks. C, H, and N elemental analyses were carried out on a Perkin–Elmer 240 instrument and Cu analysis was performed on an atomic absorption spectrophotometer, Model Z-8000. Variable-temperature magnetic susceptibilities of micro-crystalline powder sample were measured in a magnetic field 1 K Oe from 2.00 to 330 K on a SQUID magnetometer and the data were corrected for the magnetization of the sample holder and for the diamagnetic contributions of the complex which were estimated from Pascal's constants.

2.4. X-ray crystallographic analysis of the complexes

A single crystal of the complex with appropriate dimension $0.19 \times 0.13 \times 0.10$ mm^3 was chosen under an optical microscope and subsequently glued to the tip of a glass fiber. The

Table 1. Crystal data and structural refinement.

Empirical formula	C$_{15}$H$_{10}$Cu$_2$I$_3$N$_4$
Crystal system	Monoclinic
Space group	*P2$_1$/n*
Unit cell dimensions (Å,°)	
a	8.033(6)
b	14.152(11)
c	16.682(13)
β	103.932(7)
Volume (Å^3), *Z*	1841(2), 4
Calculated density (g cm^{-3})	2.721
Absorption coefficient (mm^{-1})	7.346
Final *R* indices [$I > 2\sigma(I)$]	$R_1 = 0.0415$, $wR_2 = 0.1229$
Goodness-of-fit on F^2	1.056
Largest difference peak and hole (e Å^{-3})	1.692 and −1.621

Table 2.   Selected bond lengths (Å) and angles (°).

Cu1–N2	1.958(5)	Cu1–N4	2.048(6)	Cu1–N1	2.089(6)
Cu1–I1	2.5277(16)	Cu1–I2	2.924(2)	Cu2–I2	2.519(2)
Cu2–I3	2.5603(19)	Cu2–I3A	2.5610(17)		
N2–Cu1—N4	77.3(2)	N2–Cu1–N1	159.25(15)	N4–Cu1–N1	155.4(2)
N2–Cu1–I1	155.25(15)	N4–Cu1–I1	99.75(17)	N1—Cu1–I1	99.33(15)
N2–Cu1–I2	91.76(15)	N4–Cu1—I2	94.29(16)	N1–Cu1–I2	94.07(15)
I1–Cu1–I2	108.98(4)	I2–Cu2–I3	122.19(5)	I2–Cu2–I3A	121.63(6)
I3–Cu2–I3A	115.80(7)				

Symmetry codes: I3A: $-x$, $-y+1$, $-z$.

determination of the crystal structure at 25 °C was carried out on an X-ray diffractometer (Bruker Smart-1000 CCD) using graphite-monochromated Mo-Kα radiation ($\lambda = 0.71073$ Å). Corrections for L_p factors were applied and all non-hydrogen atoms were refined with isotropic thermal parameters. Hydrogens were placed in calculated positions and refined as riding. The programs for structure solution and refinement were SHELXS-97 and SHELXL-97, respectively. Crystallographic data collection and structural refinement parameters for the complex are summarized in table 1 and table 2 gives the coordination bond lengths and associated angles.

3. Results and discussion

3.1. *Synthesis*

To obtain the product, the order of reactant addition is very important. Before sodium iodide was added, 2-(1H-pyrazol-1-yl)-1,10-phenanthroline and hydrated copper perchlorate must be mixed together and the molar ratio of the ligand 2-(1H-pyrazol-1-yl)-1,10-phenanthroline to Cu^{II} must be no less than one in order for Cu^{II} to be coordinated by 2-(1H-pyrazol-1-yl)-1,10-phenanthroline and prevent sediment of cuprous iodide. Acetonitrile was useful to dissolve 2-(1H-pyrazol-1-yl)-1,10-phenanthroline.

3.2. *Structure description*

Figure 1 shows the coordination diagram with the atom numbering scheme. The data of table 2 display that the Cu1 bond lengths are from 1.958(5) to 2.924(2) Å and the associated angles are from 77.3(2) to 159.25(15)°. Cu1 assumes a distorted square pyramidal geometry due to its Addison parameter $\tau = (\alpha - \beta)/60 = 0.06$; I2 is located at the apex of the pyramid; and the other four coordinated groups lie on the square base. The coordination bond lengths of Cu2 are 2.519(2)–2.5610(17) Å and the associated angles are 115.80(7)–122.19(5)°, which indicate that Cu2 is in a distorted trigonal planar coordination.

Based on charge neutrality and the preference of monovalent Cu^I for a trigonal planar geometry [47, 48] rather than square pyramidal, Cu2 should be monovalent and Cu1 divalent. Figure 1 also shows that Cu^{II} and Cu^I are bridged by an iodide. Cu^I-iodide bonds are very common [37–41], whereas Cu^{II}-iodide bonds [42–46] are very limited because of the reducing nature of iodide; it is rare for mixed valence Cu^{II} and Cu^I to be bridged by iodide. In the complex, the separation of the two Cu^{II} ions is 11.402 Å. The non-hydrogen

atoms of 2-(1H-pyrazol-1-yl)-1,10-phenanthroline and Cu1 atom define a plane within 0.0822 Å with a maximum deviation of −0.1911(23) Å for Cu1. In the crystal, there is a slipped π–π stacking [49] between adjacent complexes as shown in figure 2, which involves 1,10-phenanthroline and its symmetry-related counterpart, with an interplanar distance of
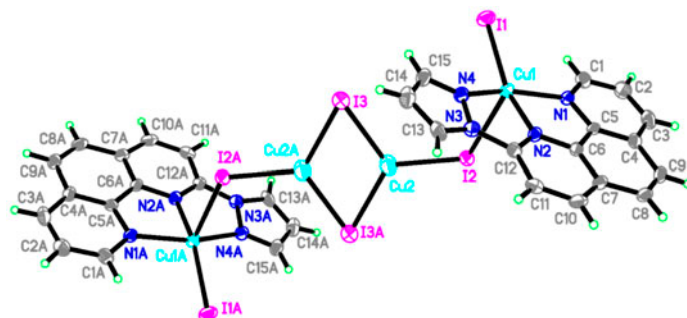

Figure 1. Coordination diagram of the complex with atom numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and the size of hydrogens is arbitrary.
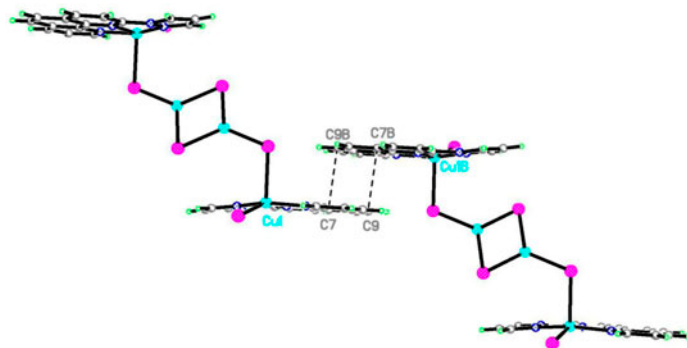

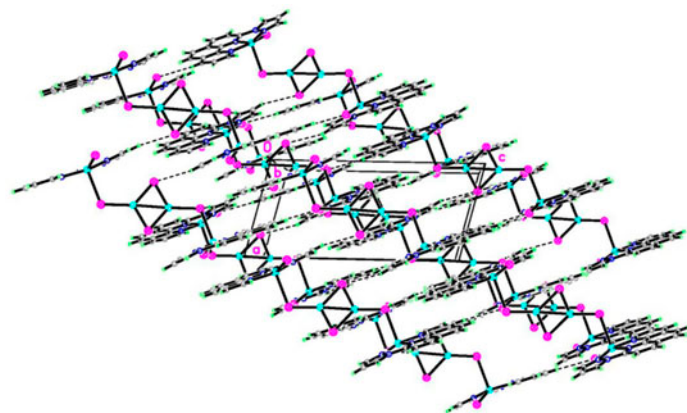Figure 2. PS-1 π–π stacking between the adjacent complexes.

Figure 3. The supramolecular 3-D stacking structure.

3.307(3) Å, dihedral angle 0.0°, and Cu1···Cu1B separation of 9.634 Å. In the π–π stacking, there are two pairs of nonbonded atoms with interatomic distances less than the sum of the default contact radii [50], C7···C9B, 3.393(10) and C9···C7B, 3.393(10) Å. The π–π stacking resulted in the formation of a supramolecular 1-D chain along the c axis and weak intermolecular forces among the chains led to the supramolecular 3-D stacking structure as shown in figure 3.

3.3. *Magnetic study*

The variable-temperature (2.00–330 K) magnetic susceptibility of the present complex is shown in figure 4, where χ_M is the molar magnetic susceptibility per binuclear Cu^{II} unit and μ_{eff} is the magnetic moment per binuclear Cu^{II} unit. Between the temperature of 330 and 50 K, the μ_{eff} value stays between 2.64 and 2.65 B.M., or $\chi_M T$ ranges from 0.87 cm^3 to 0.88 cm^3 K M^{-1}. The values are very close to those of the isolated binuclear Cu^{II} ions at room temperature ($\mu_{eff} = 2.69$ B.M., or $\chi_M T = 0.90$ cm^3 K M^{-1}, when $g = 2.2$). Then, the μ_{eff} value decreases sharply to 2.33 B.M. at 2.00 K, which indicates that there is a weak antiferromagnetic interaction between adjacent Cu^{II} ions. From the crystal structure, there are two magnetic coupling pathways between Cu^{II} ions, the bonded magnetic coupling pathway in the binuclear unit, and the π–π stacking magnetic coupling pathway. On the bonded magnetic coupling pathway of the binuclear unit, the magnetic interaction between Cu1 and Cu1A should be through Cu1↔I2↔Cu2↔I3(I3A)↔Cu2A↔I2A↔Cu1A pathway. Because these atoms are not located in a plane and there is no conjugated π system among the atoms and the separation of Cu1···Cu1A is 11.402 Å, the magnetic interaction should be very weak [51]. For the π–π stacking magnetic coupling pathway, the present π–π stacking system is very similar to the reported π–π stacking systems [31, 34–36, 52, 53] and the magnetic exchange interaction should take place in the adjacent π–π stacking binuclear Cu^{II} ions. Therefore, Bleaney-Bowers binuclear Cu^{II} equation (2), which was obtained based on isotropic Hamiltonian as presented as equation (1), was used to fit the variable-temperature magnetic susceptibility data.
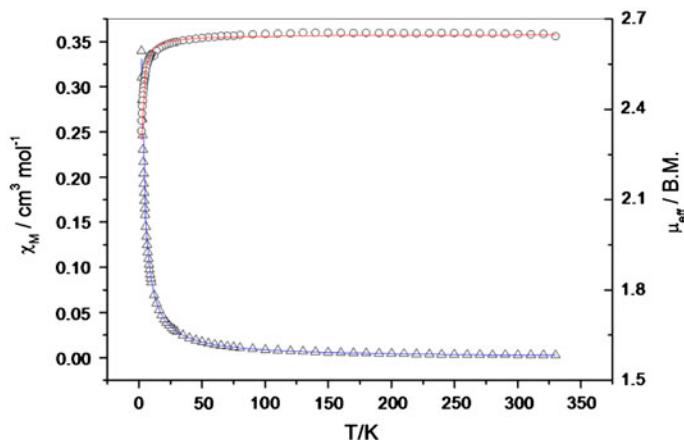

Figure 4. Plots of χ_M (the open triangle for the experimental data and the blue curve for the fitting values) and μ_{eff} (the open circle for the experimental data and the red curve for the fitting value) *vs*. T for the complex (see http://dx.doi.org/10.1080/00958972.2013.825370 for color version).
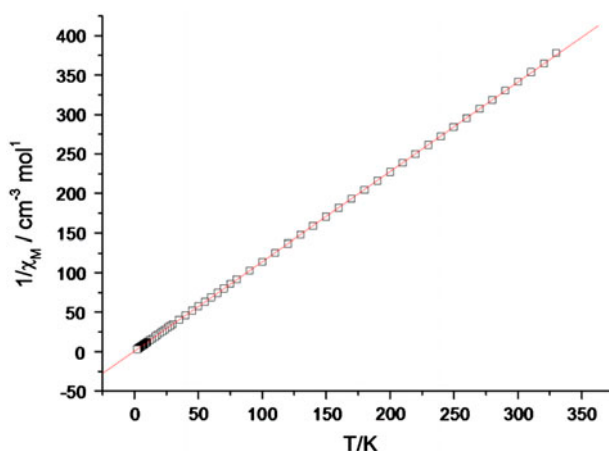
Figure 5. Thermal variation of the reciprocal susceptibility (open square for experimental data).

$$\hat{H} = -2J\hat{S}_1 \cdot \hat{S}_2 \tag{1}$$

$$\chi = \frac{2Ng^2\beta^2}{\kappa T}[3 + \exp(-2J/\kappa T)]^{-1} + N_\alpha \tag{2}$$

Equation (2) gave a good fit for the experimental data as shown in figure 3 and the relevant fitting parameters are $g = 2.16$, $2J = -1.16\,\text{cm}^{-1}$, and $R = \Sigma(\chi_{\text{obsd}} - \chi_{\text{Calcd}})^2/(\chi_{\text{obsd}})^2 = 7.0 \times 10^{-5}$. The fitting for the variable-temperature susceptibility data with Curie-Weiss formula gave Weiss constant $\theta = -0.51\,\text{K}$ as shown in figure 5. The values $2J = -1.16\,\text{cm}^{-1}$ and $\theta = -0.51\,\text{K}$ indicate that there are weak antiferromagnetic interactions between adjacent Cu^{II} ions. The present weak antiferromagnetic interaction should be attributed to π–π stacking, similar to the reported π–π stacking systems [31, 34–36, 51, 53].

4. Conclusions

A mixed-valence Cu^{I}Cu^{II} tetranuclear complex with an iodide bridge and 2-(1H-pyrazol-1-yl)-1,10-phenanthroline as the terminal ligand has been synthesized. The formation of its Cu^{II}-iodide bond and the formation of the bridged Cu^{II}-I-Cu^{I} is rare in Cu^{II} complexes with iodide as bridge. In the crystal, there is a π–π stacking between adjacent complexes. The magnetism of the complex shows a weak antiferromagnetic interaction between adjacent Cu^{II} ions, which may be ascribed to its π–π stacking magnetic coupling pathway. This work benefits understanding the magnetic coupling mechanism of π–π stacking and may also be of benefit to further explore the bonding of Cu^{II}-iodide.

Supplementary material

The ^{1}H NMR spectrum of 2-(1H-pyrazol-1-yl)-1,10-phenanthroline is provided. CCDC 927495 contains detailed information of the crystallographic data for this article; these data

can be obtained free of charge from the Cambridge Crystallographic Data Center via http://www.ccdc.cam.ac.uk/data_request/cif.

Acknowledgments

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