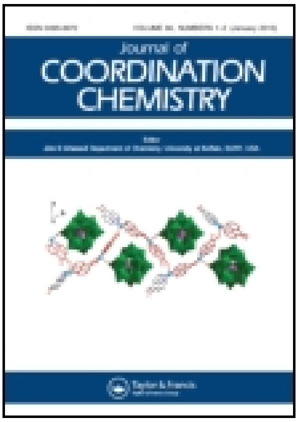

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Intermolecular interaction and magnetic coupling mechanism of a 2-D mixed-valence Cu^{II}Cu^{I} coordination polymer containing $\mu_{1,1,3}$-SCN bridge

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A 2-D Cu^{I}Cu^{II} mixed oxidation state coordination polymer, [Cu^{II}Cu^{I}($\mu_{1,3}$-SCN)$_2$($\mu_{1,1,3}$-SCN)(PhenE)]$_n$ (PhenE: 2-ethoxy-1,10-phenanthroline), has been prepared and its crystal structure determined by X-ray crystallography. In the polymer, Cu^{II} is a distorted trigonal bipyramidal geometry and Cu^{I} has distorted tetrahedral coordination. Thiocyanate bridges in two modes, $\mu_{1,3}$-SCN and $\mu_{1,1,3}$-SCN, resulting in a 2-D coordination sheet. The crystal structure analysis shows that there is a splipped π–π stacking in the sheet. The fitting for the variable-temperature magnetic susceptibility data gave the magnetic coupling constant $2J = -2.72\,\text{cm}^{-1}$ and $zJ' = -2.07\,\text{cm}^{-1}$. The magnetic interaction may be mainly ascribed to intermolecular π–π magnetic coupling.

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

1. Introduction

Molecular magnetism has attracted attention and major advances have been made in description and application as new molecular-based materials [1, 2]. Research has concentrated on systems where coupling spin-carriers (or paramagnetic centers) are linked by bridging ligands with magnetic interactions through bond exchange [3–7]. In magnetic coupling, intermolecular interactions also play a role. For example, some authors attributed strong ferromagnetic order to π–π stacking interactions, [8], and other authors found π–π stacking interactions led to a strong anti-ferromagnetic interaction between spin-carriers [9–11]. Strong anti-ferromagnetic coupling between Cu^{II} ions through O–H$\cdots$O hydrogen bonding has also been reported [12]. Intermolecular forces should be a factor in magnetic coupling properties. However, not much work is published on π–π stacking

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*Corresponding author. Email: sdnuccm@163.com systems [8–11], hydrogen bonding [13–20], and X–H$\cdots\pi$ interactions [16]. Most articles only deal with radicals [13, 20] or complexes [11, 21–24] with radicals as ligands. Factors that dominate the magnetic coupling properties have not been clearly understood. Thus, it is important to synthesize complexes with intermolecular interactions and to study the factors that dominate magnetic coupling mechanism, an area to which our attention has been given [25–30].

2-Ethoxy-1,10-phenanthroline possesses strong chelated coordination and large conjugation plane, which may be useful to form complexes with strong π–π stacking and relevant magnetic coupling pathway. So far no crystal structure of a complex with 2-ethoxy-1,10-phenanthroline as ligand has been published. Thiocyanate generally functions as $\mu_{1,3}$-SCN bridge [28, 31] and it rarely acts as $\mu_{1,1}$-SCN [32–34] or $\mu_{1,1,3}$-SCN [35–41] bridges. Herein, we report a 2-D mixed-valence $Cu^{I}Cu^{II}$ coordination polymer with thiocyanate as both $\mu_{1,3}$-SCN and $\mu_{1,1,3}$-SCN bridges and its magnetic coupling mechanism.

2. Experimental

2.1. *Materials*

2-Ethoxy-1,10-phenanthroline was synthesized by the following method. 2-Chloro-1,10-phenanthroline (2.08 g, 9.69 mM) and K_2CO_3 (4.6 g, 33.3 mM) were dissolved in 30 mL ethanol and refluxed for four days, and precipitate appeared after a small amount of broken ice was added into the reaction solution; the product (2.11 g, 9.41 mM) was obtained after the precipitate was washed with water until the filtrate was neutral and then dried. All other chemicals are analytical grade and used without purification.

2.2. *Preparation of complex*

Methanol solution (5 mL) of $Cu(ClO_4)_2 \cdot 6H_2O$ (0.0733 g, 0.198 mM) was added into 10 mL acetonitrile solution of 2-ethoxy-1,10-phenanthroline (0.0502 g, 0.224 mM) and then 5 mL methanol solution of NaSCN (0.0158 g, 0.195 mM) was added. The mixed solution was stirred for a few minutes. Deep green single crystals were obtained after the filtrate was allowed to slowly evaporate at room temperature for one week. IR (cm^{-1}): 2126(s), 2112(s), 1611(m), 2810, 1589(m), 1511(m), 1499(m), 1288(m), 1028(m), 850(w). Elemental Anal. Calcd for $C_{17}H_{12}Cu_2N_5OS_3$ (Fw 525.58): C, 38.85; H, 2.30; N, 13.33; Cu, 24.18%. Found: C, 39.12; H, 2.65; N, 13.01; Cu, 24.65%.

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 content was obtained on an atomic absorption spectrophotometer, Model Z-8000. Variable-temperature magnetic susceptibilities of microcrystalline 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 magnetization of the sample holder and 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 dimensions $0.32 \times 0.32 \times 0.18$ mm^3 was chosen under an optical microscope and subsequently glued to the tip of a glass fiber. The 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 anisotropic 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. The pertinent 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. *Structure description*

Figure 1 shows the coordination diagram with the atom numbering scheme. The data of table 2 display that the coordination bond lengths of Cu1 atom range from 1.9428(18) Å to 2.5032(8) Å and associated angles change from 80.78(8)° to 172.55(8)°. Obviously, Cu1 assumes a distorted trigonal bipyramidal geometry; the coordination bond lengths of Cu2 cover 1.912(2)–2.6340(9) Å and associated angles are 96.61(7)–129.20(9)°, which indicate that Cu2 is a distorted tetrahedral coordination environment. Figure 1 also shows that S1C15N3 thiocyanate is a $\mu_{1,1,3}$-bridge, connecting three coppers. Compared with complexes having $\mu_{1,3}$-SCN bridges, only a few complexes [35–41] deal with $\mu_{1,1,3}$-SCN bridges. Due to thiocyanate acting as two kinds of bridges, a 2-D sheet structure along the (101) plane is formed.

On the basis of charge neutrality, the complex is in a mixed-valence state and its chemical formula is $[\mathrm{Cu^{II}Cu^{I}}(\mu_{1,3}\text{-SCN})_2(\mu_{1,1,3}\text{-SCN})(\mathrm{PhenE})]_n$. Based on the preference of Cu^{I} for a tetrahedral [42–45] rather than trigonal bipyramidal geometry, Cu2 should be Cu^{I} and Cu1, Cu^{II}. In figure 1, the separations of the adjacent divalent Cu^{II} ions that are connected by bonds are 10.970 Å for Cu1 and Cu1B, 7.734 Å for Cu1 and Cu1E and 10.086 Å for Cu1 and Cu1D. The non-hydrogen atoms of 1,10-phenanthroline and Cu1 define a plane within 0.0342 Å with a maximum deviation of 0.0988(10) Å for Cu1. In the

Table 1. Crystal data and structural refinements.

Empirical formula	$C_{17}H_{12}Cu_2N_5OS_3$
Crystal system	Monoclinic
Space group	$P2_1/n$
Unit cell dimensions (Å, °)	
a	7.5329(12)
b	14.654(2)
c	17.903(3)
β	99.458(2)
Volume (Å^3), Z	1949.3(5), 4
Calculated density (g cm^{-3})	1.791
Absorption coefficient (mm^{-1})	2.522
Final R indices [I $2\sigma(I)$]	$R_1 = 0.0319$, $wR_2 = 0.0767$
Goodness-of-fit on F^2	0.939
Largest difference peak and hole (e Å^{-3})	0.525 and −0.271

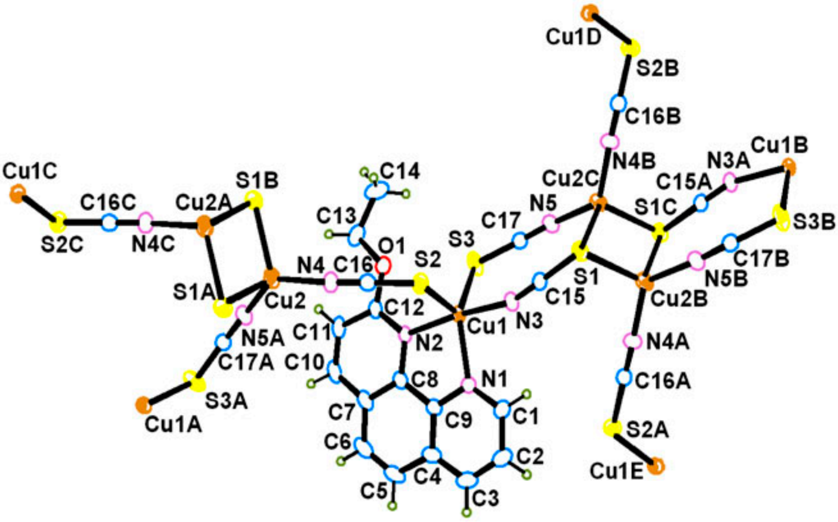

Figure 1. Coordination of the complex with atom numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and the size of H is arbitrary.

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

Cu1–N3	1.9428(18)	Cu1–N2	2.0097(17)	Cu1–N1	2.0898(19)
Cu1–S3	2.3947(8)	Cu1–S2	2.5032(8)	Cu2–N4	1.912(2)
Cu2–N5^{i}	1.925(2)	Cu2–S1^{ii}	2.3830(8)	Cu2–S1^{i}	2.6340(9)
Cu2–Cu2^{iii}	2.8953(8)				
N3–Cu1–N2	172.55(8)	N3–Cu1–N1	92.90(8)	N2–Cu1–N1	80.78(8)
N3–Cu1–S3	94.17(6)	N2–Cu1–S3	92.82(6)	N1–Cu1–S3	127.55(6)
N3–Cu1–S2	84.05(7)	N2–Cu1–S2	94.80(6)	N1–Cu1–S2	113.39(6)
S3–Cu1–S2	119.02(3)	N4–Cu2–N5^{i}	129.20(9)	N4–Cu2–S1^{ii}	110.76(7)
N5^{i}–Cu2–S1^{ii}	106.16(7)	N4–Cu2–S1^{i}	102.30(7)	N5^{i}–Cu2–S1^{i}	96.61(7)
S1^{ii}–Cu2–S1^{i}	109.71(2)				

Note: Symmetry codes: i: $x-½$, $-y+3/2$, $z-½$; ii: $-x+½$, $y+½$, $-z+½$; iii: $-x$, $-y2$, $-z$.

2-D sheet, there is a splipped π–π stacking interaction [46] with name PS-1 as shown in figure 2, which involves 1,10-phenanthroline and its symmetry-related counterpart, with an interplanar distance of 3.424 Å and dihedral angle 0.0°. In PS-1 system, there are five pairs of non-bonded atoms with separation less than 3.6 Å, namely, C3···C7A and C7···C3A, 3.463(4) Å; C4···C4A, 3.597(4) Å; C5···C9A and C9···C5A, 3.438(4) Å. In PS-1, the separation of Cu1 and Cu1A is 8.7564(10) Å. The π–π stacking may solidify the crystal.

3.2. *Magnetic study*

The variable-temperature (2.00–330 K) magnetic susceptibility of the present complex is shown in figure 3, 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. The μ_{eff} value at 330 K is 2.59 B.M., larger than that of the isolated binuclear Cu^{II} ions (2.45 B.M., $g=2$) at room temperature. The value of μ_{eff} decreases very slowly with temperature until about 30 K and then it

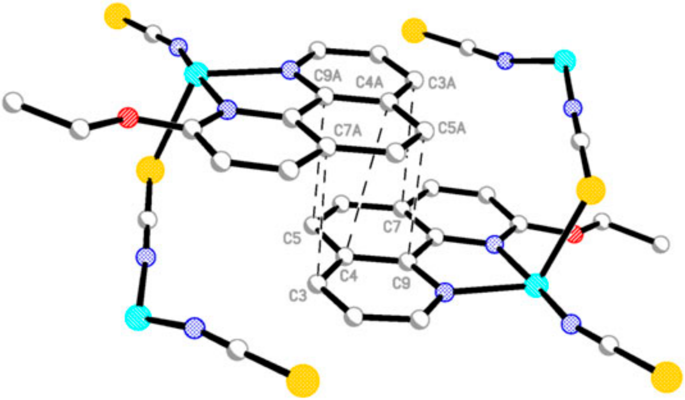

Figure 2. PS-1 π–π stacking in the 2-D sheet (symmetry code: $-x$, $1-y$, $-z$).

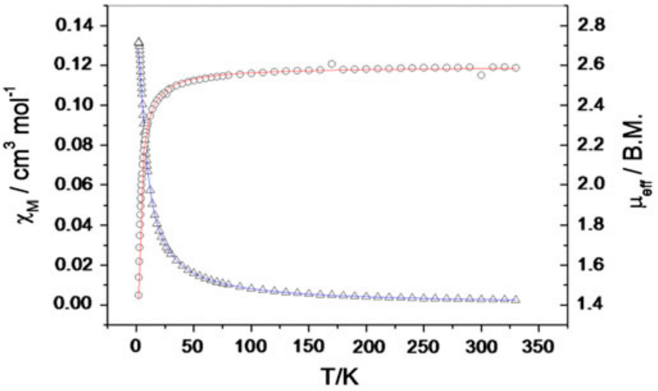

Figure 3. Plots of χ_M (the open triangle for the experimental data and the blue curve for the fitting values) and μ_{eff} (the open circle for the experimental data and the red curve for the fitting value) vs. T for the complex.

starts to decrease sharply to 1.45 B.M. at 2.00 K, which indicates that there is a weak antiferromagnetic interaction between adjacent Cu^{II} ions. At first, the formulas of a uniform 1-D Cu^{II} chain and alternating 1-D Cu^{II} chain were used to fit the data of the variable-temperature magnetic susceptibility, but it failed. Then the modified Bleaney–Bowers binuclear Cu^{II} equations (2) and (3) [27], based on isotropic Hamiltonian as presented as equation (1) were used to fit the experimental susceptibility data and it fits well.

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

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

$$\chi_M = \frac{\chi}{1 - \frac{zJ'\chi}{Ng^2\beta^2}} \tag{3}$$

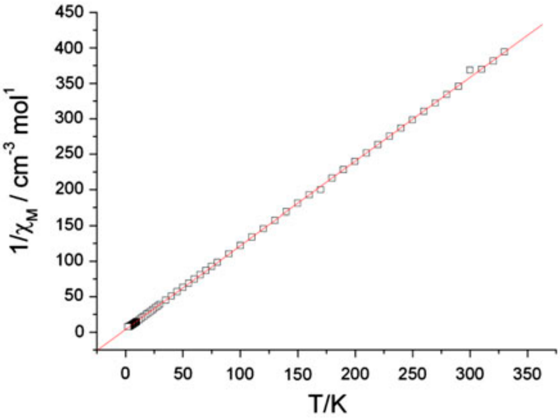

Figure 4. Thermal variation of the reciprocal susceptibility (open square for experimental data).

Equations (2) and (3) gave a good fit to the experimental data as shown in figure 3, and the relevant fitting parameters are as follows: $g = 2.12$, $2J = -2.72\,\mathrm{cm}^{-1}$, $zJ' = -2.07\,\mathrm{cm}^{-1}$, and $R = \Sigma(\chi_{\mathrm{obsd}} - \chi_{\mathrm{calcd}})^2/(\chi_{\mathrm{obsd}})^2 = 8.0 \times 10^{-5}$. The fitting for the variable-temperature susceptibility data with Curie–Weiss formula gave Weiss constant $\theta = -3.01\,\mathrm{K}$ as shown in figure 4. The values $2J = -2.72\,\mathrm{cm}^{-1}$, $zJ' = -2.07\,\mathrm{cm}^{-1}$, and $\theta = -3.01\,\mathrm{K}$ indicate that there are weak antiferromagnetic interactions between adjacent $\mathrm{Cu^{II}}$ ions. From the crystal structure, there are three magnetic coupling pathways among adjacent $\mathrm{Cu^{II}}$ ions, which include two bonded magnetic coupling pathways and one unbonded magnetic coupling pathway. The two bonded magnetic coupling pathways involve the uniform 1-D chain pathway [$\mathrm{Cu^{II}}$ (Cu1) double thiocyanate bridges $\mathrm{Cu^{I}}$ (Cu2B or Cu2C) single thiocyanate bridge $\mathrm{Cu^{II}}$ (Cu1D or Cu1E)] and the alternating 1-D chain pathway [$\mathrm{Cu^{II}}$ (Cu1B) double thiocyanate bridges double $\mathrm{Cu^{I}}$ ions (Cu2B and Cu2C) double thiocyanato bridge anions $\mathrm{Cu^{II}}$ (Cu1) single thiocyanate bridge double $\mathrm{Cu^{I}}$ (Cu2 and Cu2A) single thiocyanate bridge $\mathrm{Cu^{II}}$ (Cu1C)] as shown in figure 1. The one unbonded magnetic coupling pathway is PS-1 π–π stacking pathway. In the two bonded magnetic coupling pathways, the magnetic exchange interaction should be propagated by the delocalized π orbital of the thiocyanate bridge and d orbital of $\mathrm{Cu^{I}}$. However, the bridged thiocyanates are not located in a plane. Furthermore, for each pair of coupling $\mathrm{Cu^{II}}$ ions, there is an interval of at least two thiocyanates and a $\mathrm{Cu^{I}}$, which limits electron delocalization [47]. Therefore, the magnetic interactions from the two bonded magnetic coupling pathways should be very weak and ignored, and the obtained fitting values should be mainly attributed to PS-1 magnetic coupling pathway.

4. Conclusions

A 2-D mixed-valence $\mathrm{Cu^{I}Cu^{II}}$ coordination polymer with thiocyanate as $\mu_{1,3}$-SCN and $\mu_{1,1,3}$-SCN mixed bridges and 2-ethoxy-1,10-phenanthroline as terminal ligand has been synthesized. Its crystal structure allows interpretation of three magnetic coupling pathways between adjacent $\mathrm{Cu^{II}}$ ions, two of which deal with adjacent $\mathrm{Cu^{II}}$ ions linked by thiocyanate bridges, and the other one involves intermolecular π–π stacking. The analysis of the magneto-structural correlation indicates that the weak antiferromagnetic interaction of the coordination polymer should be mainly attributed to the intermolecular π–π stacking. This work benefits understanding magnetic coupling mechanism of π–π stacking and the bridge coordination modes of thiocyanate.

Supplementary material

CCDC 893106 contains detailed information of the crystallographic data for this article, and these data can be obtained free of charge from the Cambridge Crystallographic Data Center via http://www.ccdc.cam.ac.uk/data_request/cif.

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