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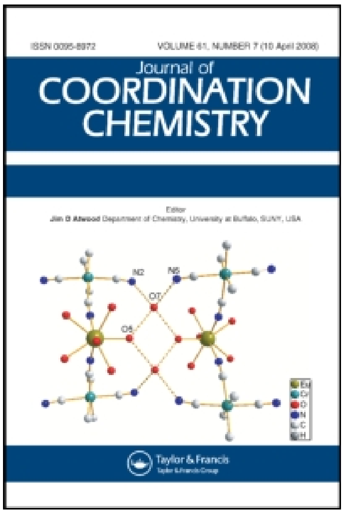

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Shu-Ping Wang[a]; Jing Chen[a]; Dong-Zhao Gao[a]; You Song[b]; Qing-Mei Wang[a]; Dai-Zheng Liao[a]; Zong-Hui Jiang[ab]; Shi-Ping Yan[a]
[a] Department of Chemistry, Nankai University, Tianjin 300071, China [b] State Key Laboratory of Coordination Chemistry, Nanjing University, Nanjing 210093, China

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# Ferromagnetic interactions in a Zn(II)–nitronyl nitroxide complex with dicyanoaurate(I) bridges

SHU-PING WANG†, JING CHEN†, DONG-ZHAO GAO†,
YOU SONG‡, QING-MEI WANG†, DAI-ZHENG LIAO†,
ZONG-HUI JIANG*†‡ and SHI-PING YAN†

†Department of Chemistry, Nankai University, Tianjin 300071, China
‡State Key Laboratory of Coordination Chemistry, Nanjing University,
Nanjing 210093, China

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A two-dimensional Zn(II)-nitronyl nitroxide radical complex, $Zn(NIT4Py)_2[Au(CN)_2]_2$ (NIT4Py = 2-(4′-pyridyl)-4,4,5,5-tetramethylimidazoline-1-oxyl-3-oxide) was synthesized and characterized by elemental analyses, IR spectroscopy and single-crystal X-ray diffraction methods. The complex is monoclinic, space group $C2/c$, with $a = 26.294(9)$, $b = 7.167(2)$, $c = 18.532(6)$ Å, $\beta = 111.758(5)^\circ$, $V = 3243.6(18)$ Å$^3$, $Z = 4$, and $R_1$ $[I > 2\sigma(I)] = 0.0348$, and has an infinite two-dimensional network motif. Magnetic studies showed ferromagnetic coupling between two radicals through Zn(II).

*Keywords:* Nitronyl nitroxide radical; Zinc(II); Dicyanoaurate(I); Two-dimensional complex; Ferromagnetic interaction

## 1. Introduction

The design and synthesis of multidimensional molecular-based magnetic materials with ferromagnetic ordering is one of the major challenges in molecular materials research [1]. For a magnetic system, macroscopic magnetic properties are strongly dependent on the dimensionality of the structure and on the magnitude of the exchange interaction between the spin carriers. Therefore, an effective strategy in designing molecular-based magnetic materials is to enhance the dimensionality of the magnetic coupling system.

Nitronyl nitroxide radicals have attracted much interest during the past decade because they have been envisaged as active building blocks for the construction of coordination compounds exhibiting spontaneous magnetization or interesting optical properties [2, 3]. In the field of metal–radical magnetic materials, pseudohalide, dicyanide and $[Ag(CN)_2]^-$ anions are extensively used as bridges to assemble extended coordination geometries. High spin clusters [3], one-dimensional compounds [4, 5],

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*Corresponding author. Email: coord@nankai.edu.cn

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two-dimensional [6] and three-dimensional systems [7] have been reported. It is possible to build up high-dimensionality magnetic systems by choosing appropriate bridges. $[Au(CN)_2]^-$ has been used as a bridge between metals in a one-dimensional transition metal–radical complex [8]. However, reports on high-dimensionality transition metal–radical complexes with the dicyanoaurate(I) bridge are scarce. Therefore, herein a novel two-dimensional Zn(II)-radical complex with dicyanoaurate(I) bridges is reported.

Several diamagnetic metal complexes with organic radical ligands have shown antiferro- or ferromagnetic interactions operative through the diamagnetic ions (such as Ti(IV) [9], Cu(I), Ag(I) [10] and Hg(II) [11]). In these complexes, the metal ions have an active role in aiding the exchange coupling between the radicals, promoting superexchange interactions between organic molecules. There are a few reports concerning Zn(II), Cd(II) organic radical complexes, but most of them show antiferromagnetic interactions [12–15]. In the present article, the two-dimensional complex $Zn(NIT4Py)_2[Au(CN)_2]_2$ exhibits a ferromagnetic interaction.

## 2. Experimental

### 2.1. *Physical measurements*

Elemental analyses for carbon, hydrogen and nitrogen were carried out on a Perkin-Elmer 240 instrument. IR spectra were recorded on a Shimadzu 408 spectrophotometer in the 4000–600 $cm^{-1}$ range, using KBr pellets. Variable temperature susceptibility measurements were carried out with a SQUID MPMS XL7 magnetometer in the temperature range 2.0–300 K at a magnetic field of 2000 G. Molar susceptibilities were corrected for the sample holder and diamagnetic contributions of all constituent atoms, using Pascal's constants.

### 2.2. *Materials and synthesis*

All starting materials were of analytical grade and used as purchased without further purification. NIT4Py was prepared according to reported methods [16]. An aqueous solution of $K[Au(CN)_2]$ (0.2 mmol) was added dropwise to a methanol solution of $Zn(ClO_4)_2 \cdot 6H_2O$ (0.1 mmol) and NIT4Py (0.2 mmol) at room temperature. The mixed solution was stirred for 4 h and filtered. Deep blue-green, single crystals suitable for X-ray structure analysis were grown at room temperature by the slow evaporation of the filtrate. Note: Cyanide salts are toxic and should be handled with caution! IR ($cm^{-1}$): $\nu$(C–N), 2150; $\nu$(N–O), 1368. Anal. Calcd for $C_{28}H_{32}Au_2ZnN_{10}O_4$ (%): C, 32.59; H, 3.13; N, 13.57. Found: Cm 32.42; H, 3.40; N, 13.58.

### 2.3. *X-ray crystallography*

A single crystal of the title complex was mounted on a Bruker Smart 1000 CCD diffractometer using graphite-monochromated Mo-$K\alpha$ radiation ($\lambda = 0.71073$ Å). Data were collected at room temperature by the $\varphi$–$\omega$ scan mode. Unit cell parameters were determined from $2.37^\circ \leq \theta \leq 26.41^\circ$ for the complex. Details of data collection, crystallographic data and structure refinement parameters are summarized in table 1.

Table 1. Crystallographic data for the complex.

| | |
|---|---|
| Empirical formula | $C_{28}H_{32}N_{10}O_4Au_2Zn$ |
| Formula weight | 1031.94 |
| Temperature (K) | 293(2) |
| Wavelength (Å) | 0.71073 |
| Crystal system, space group | Monoclinic, $C2/c$ |
| Unit cell dimensions | $a = 26.294(9)$ Å<br>$b = 7.167(2)$ Å, $\beta = 111.758(5)^\circ$<br>$c = 18.532(6)$ Å |
| Volume (Å$^3$) | 3243.6(18) |
| $Z$, calculated density (Mg m$^{-3}$) | 4, 2.113 |
| Absorption coefficient (mm$^{-1}$) | 9.807 |
| $F(000)$ | 1960 |
| Crystal size (mm$^3$) | $0.22 \times 0.20 \times 0.18$ |
| $\theta$ range for data collection (°) | 2.37 to 26.41 |
| Limiting indices | $-32 \leq h \leq 14$, $-8 \leq k \leq 8$, $-23 \leq l \leq 23$ |
| Reflections collected/unique | 8954/3300 [$R_{\text{int}} = 0.0569$] |
| Completeness to $\theta = 25.00$ (%) | 99.9% |
| Absorption correction | Semi-empirical from equivalents |
| Max. and min. transmission | 1.0000 and 0.4919 |
| Refinement method | Full-matrix least-squares on $F^2$ |
| Data/Restraints/Parameters | 3300/0/210 |
| Goodness-of-fit on $F^2$ | 0.967 |
| Final $R$ indices [$I > 2\sigma(I)$] | $R_1 = 0.0348$, $wR_2 = 0.0660$ |
| $R$ indices (all data) | $R_1 = 0.0564$, $wR_2 = 0.0716$ |
| Largest diff. peak and hole (e Å$^{-3}$) | 1.273 and $-2.086$ |

The structure was solved with direct methods using SHELXS-97 [17]. H atoms were assigned common isotropic displacement factors and included in the final refinement by use of geometrical restraints. A full-matrix least-squares refinement on $F^2$ was carried out using SHELXL-97 [18]. Reliability factors were defined as $R_1 = \Sigma(\|F_o| - |F_c\|)/\Sigma|F_o|$, and the function minimized was $wR_2 = (\Sigma w(|F_o|^2 - |F_c|^2)^2/\Sigma w|F_o|^2)^{1/2}$, where in the least-squares calculation unit weights were used. All non-hydrogen atoms were refined anisotropically. Final atomic coordinates for all non-hydrogen atoms and equivalent thermal parameters are listed in table 2 and selected bond distances and angles are listed in table 3.

## 3. Results and discussion

### 3.1. *Crystal structure*

A perspective view of $Zn(NIT4Py)_2[Au(CN)_2]_2$ is shown in figure 1. The crystal structure consists of infinite networks, in which $Zn(NIT4Py)_2$ moieties are connected by $\mu_2$-bridging $[Au(CN)_2]^-$ ligands in the *bc* plane (figure 2).

$Zn(NIT4Py)_2[Au(CN)_2]_2$ is a neutral molecule in which each zinc atom is six-coordinated in a distorted, centrosymmetric, octahedral environment. The equatorial plane is formed by four nitrogen atoms from four $[Au(CN)_2]^-$ groups (N1, N1A, N2B, N2C). Separate Zn–N bond lengths in the basal plane are 2.227(5) and 2.157(5) Å. Axial positions are occupied by nitrogen atoms of pyridyl rings of NIT4Py radicals, with a Zn–N distance of 2.108(5) Å. Bond angles in the octahedron are slightly distorted from ideal values [87.0(2)–93.0(2)°]. The angle between the pyridyl ring and the nitroxide group is 29.3° in NIT4Py and the distance between adjacent

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters (Å$^2 \times 10^3$) for the non-hydrogen atoms.

| | $x/a$ | $y/b$ | $z/c$ | $U_{eq}$ |
|---|---|---|---|---|
| Au(1) | 2222(1) | 664(1) | 2204(1) | 33(1) |
| Zn(1) | 2500 | −2500 | 0 | 20(1) |
| C(1) | 2222(2) | 275(9) | 1138(4) | 28(1) |
| C(2) | 2231(2) | 909(9) | 3269(4) | 31(2) |
| C(3) | 3330(2) | 587(8) | 189(3) | 25(1) |
| C(4) | 3829(2) | 1443(8) | 389(4) | 28(1) |
| C(5) | 4302(2) | 434(8) | 732(3) | 24(1) |
| C(6) | 4251(2) | −1459(8) | 873(3) | 24(1) |
| C(7) | 3738(2) | −2203(8) | 665(3) | 22(1) |
| C(8) | 4845(2) | 1278(7) | 1009(3) | 22(1) |
| C(9) | 5564(2) | 3483(8) | 1474(3) | 24(1) |
| C(10) | 5796(2) | 1489(8) | 1652(3) | 26(1) |
| C(11) | 5637(3) | 4368(8) | 775(4) | 34(2) |
| C(12) | 5729(3) | 4807(10) | 2158(4) | 43(2) |
| C(13) | 6296(3) | 1088(9) | 1437(4) | 39(2) |
| C(14) | 5908(3) | 818(10) | 2476(4) | 52(2) |
| N(1) | 2226(2) | −153(7) | 555(3) | 29(1) |
| N(2) | 2251(2) | 1102(7) | 3892(3) | 32(1) |
| N(3) | 3274(2) | −1221(6) | 317(3) | 21(1) |
| N(4) | 4959(2) | 3057(7) | 1231(3) | 24(1) |
| N(5) | 5320(2) | 366(7) | 1136(3) | 28(1) |
| O(1) | 4616(2) | 4329(5) | 1211(3) | 39(1) |
| O(2) | 5370(2) | −1278(6) | 913(3) | 49(1) |

Table 3. Selected bond lengths (Å) and angles (deg) for the complex.

| | | | |
|---|---|---|---|
| Au(1)–C(2) | 1.974(7) | Au(1)–C(1) | 1.994(6) |
| Zn(1)–N(1) | 2.227(5) | Zn(1)–N(2)[b] | 2.157(5) |
| Zn(1)–N(3) | 2.108(5) | C(1)–N(1) | 1.127(7) |
| C(2)–N(2) | 1.144(8) | C(3)–N(3) | 1.335(7) |
| C(7)–N(3) | 1.347(7) | C(8)–N(4) | 1.339(7) |
| C(8)–N(5) | 1.350(7) | C(9)–N(4) | 1.516(7) |
| C(10)–N(5) | 1.497(8) | N(4)–O(1) | 1.273(6) |
| N(5)–O(2) | 1.272(6) | | |
| C(2)–Au(1)–C(1) | 177.0(3) | N(3)–Zn(1)–N(2)[b] | 89.05(18) |
| N(3)–Zn(1)–N(1) | 89.28(18) | N(3)–Zn(1)–N(2)[c] | 90.95(18) |
| N(2)[b]–Zn(1)–N(1) | 93.0(2) | N(3)–Zn(1)–N(1)[a] | 90.72(18) |
| N(2)[c]–Zn(1)–N(1) | 87.0(2) | N(1)–C(1)–Au(1) | 172.2(6) |

Symmetry transformations used to generate equivalent atoms are: (a) $-x+1/2$, $-y-1/2$, $-z$; (b) $x$, $-y$, $z-1/2$; (c) $-x+1/2$, $y-1/2$, $-z+1/2$.

Zn atoms linked by $[Au(CN)_2]^-$ bridges is 9.935 Å. The shortest radical contact within a layer is 3.869 Å (O1 $\cdots$ O2), and that between adjacent layers 3.703 Å (O2 $\cdots$ O2).

## 3.2. *Magnetic properties*

Measured $\chi_M T$ *versus* $T$ and $\chi_M^{-1}$ *versus* $T$ plots for the complex in the range 5–300 K are shown in figure 3, where $\chi_M$ is the magnetic susceptibility per $Zn(NIT4Py)_2[Au(CN)_2]_2$ unit. The $\chi_M T$ value at room temperature is 0.76 cm$^3$ K mol$^{-1}$, close to that expected for independent spins (0.75 cm$^3$ K mol$^{-1}$ for two $S_R = 1/2$). As temperature is lowered, the $\chi_M T$ value increases slowly and reaches a maximum value of 0.82 cm$^3$ K mol$^{-1}$ at 22 K, and then decreases rapidly on further lowering of temperature. From the

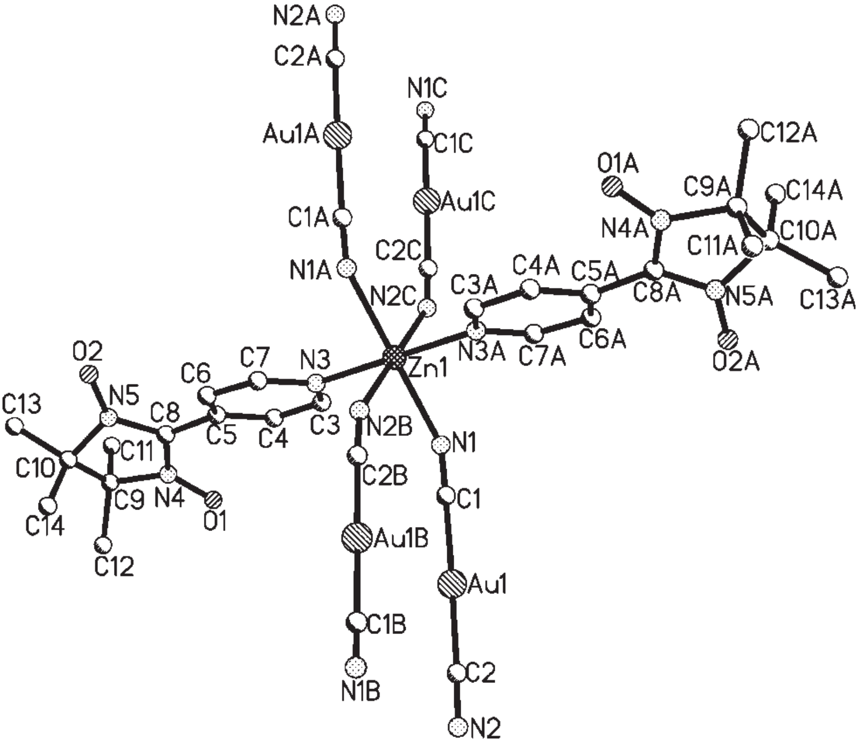

Figure 1. View of the complex with 50% thermal ellipsoids, showing the atom labeling scheme.

plot of reciprocal susceptibility ($\chi_M^{-1}$) against $T$, the complex follows the Curie–Weiss law in the range 5–300 K, with a Curie constant of $0.763\,\text{cm}^3\,\text{K}\,\text{mol}^{-1}$. The Weiss temperature is found to be $\theta = 1.25\,\text{K}$. The magnetic behavior suggests that some ferromagnetic interactions are predominant at intermediate temperatures and then weaker antiferromagnetic couplings are involved at lower temperature.

From structural features, there are two major magnetic interactions for the present system, that between NO groups through the NO(1)–Zn–NO(1A) pathway in Zn(NIT4py)$_2$ unit in the two-dimensional layer, and close contacts between NO groups of radicals (through space). Exchange interactions by these pathways are expected to be weak. To evaluate the exchange coupling constants between radicals through Zn(II), the complex was treated as a biradical system (two spin $S = 1/2$), and average magnetic interactions of nearest radicals through space, $zJ'$, as a molecular field approximation. The Hamitonian for the biradical system can be written as $\hat{H} = -2J\hat{S}_1\hat{S}_2$ and the magnetic susceptibility can be expressed as $\chi = (2Ng^2\beta^2/kT)[3 + \exp(-2J/kT)]^{-1}$. In this expression, $2J$ is the exchange coupling constant between the nitronyl nitroxide groups through Zn(II). The total magnetic susceptibility is $\chi_M = \chi\ /[1 - \chi(2zJ'/Ng^2\beta^2)]$, where $\chi_M$ represents the magnetic susceptibility per Zn(NIT4Py)$_2$[Au(CN)$_2$]$_2$ unit.

Least-squares analysis of magnetic susceptibilities (data for $T > 5\,\text{K}$) led to $g = 2$ (fixed), $J = 9.66\,\text{cm}^{-1}$, $zJ' = -1.51\,\text{cm}^{-1}$ and $R = \Sigma[(\chi_M)_{\text{obs}} - (\chi_M)_{\text{calc}}]^2/\Sigma[(\chi_M)_{\text{obs}}]^2 = 1.75 \times 10^{-4}$, indicating a weak intramolecular ferromagnetic interaction.

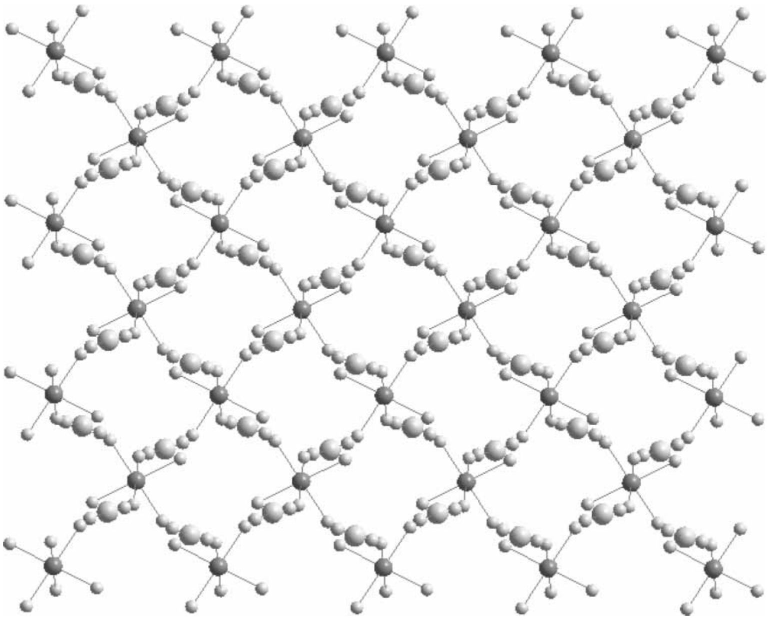

Figure 2. View of the network for Zn(NIT4Py)$_2$[Au(CN)$_2$]$_2$ along the *bc* plane. For the sake of clarity NIT4Py groups have been omitted except for pyridyl-*N*.

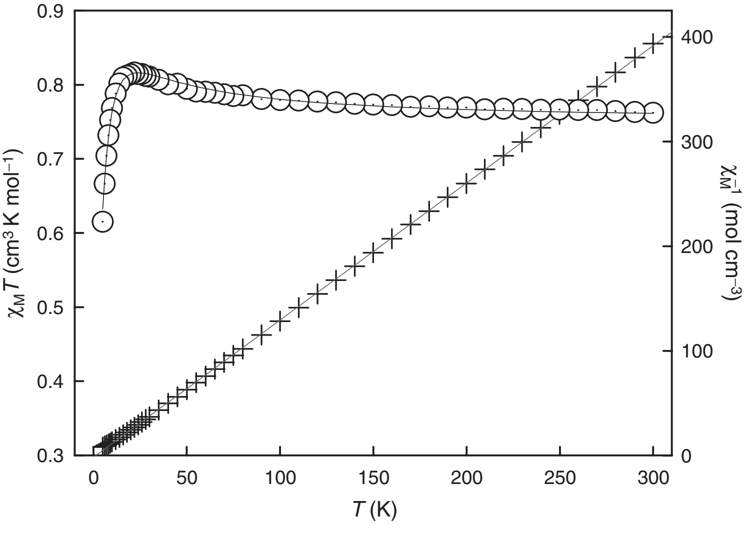

Figure 3. Plots of $\chi_M T$ (⊙) and $\chi_M^{-1}$ (+) *vs*. *T* for the complex. The solid lines correspond to the best theoretical fits.

Observed ferromagnetic coupling between radicals through diamagnetic Zn(II) may be due to the d-orbitals of zinc(II) having appropriate energy and symmetry to match with the $\pi$ orbitals of NIT4Py. This implies that the spin density of each NIT4Py $\pi$ electron is delocalized partially onto zinc(II), thus providing the main orthogonal super-exchange pathway.

In conclusion, a two-dimensional diamagnetic metal nitronyl nitroxide radical complex with dicyanoaurate(I) bridges has been synthesized. The diamagnetic metal centres and dicyanoaurate(I) bridges have been used as a platform for the construction of molecules in which the spin carriers are located around the periphery. The diamagnetic metals appear to have an active role in aiding exchange coupling between the radicals. Although only a weak ferromagnetic interaction was observed between NIT4Py radicals in the compound, it is the first example of polymeric two-dimensional Zn(II) compounds with paramagnetic nitronyl nitroxide ligands.

## Supplementary material

CCDC-226024 contains supplementary crystallographic data for this article. These data can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Centre (CCDC), 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033).

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