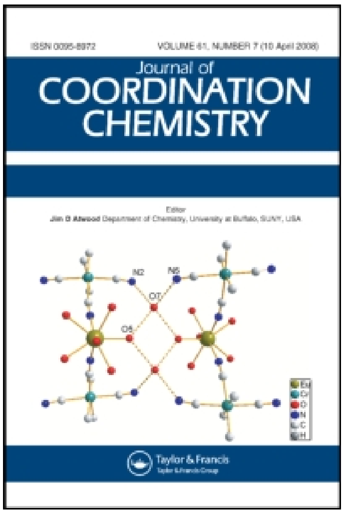

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Crystal structure and magnetic properties of a nitronyl nitroxide complex $\{[Co_2(NIT4Py)_4(fum)_2(H_2O)_4] \cdot (H_2O)_2\}$*<sub>n*

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Crystal structure and magnetic properties of a nitronyl nitroxide complex $\{[Co_2(NIT4Py)_4(fum)_2(H_2O)_4] \cdot (H_2O)_2\}_n$

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The title compound $\{[Co_2(NIT4Py)_4(fum)_2(H_2O)_4] \cdot (H_2O)_2\}_n$, where NIT4Py = 2-(4′-pyridinyl)-4,4,5,5-tetra-methylimidazoline-1-oxyl-3-oxide and fum = fumarate, was prepared and structurally characterized by X-ray diffraction. It crystallizes in triclinic, space group $P\bar{1}$ with $a = 9.8853(9)$ Å, $b = 13.2167(13)$ Å, $c = 13.8481(12)$ Å, $\alpha = 68.5400(10)^\circ$, $\beta = 73.467(2)^\circ$, $\gamma = 74.596(2)^\circ$, $V = 1587.6(3)$ Å^3, $Z = 1$, and R_1 $[I > 2\sigma(I)] = 0.0662$. The complex consists of two kinds of neutral infinite 1-D chains, in which $[Co(NIT4Py)_2(H_2O)_2]$ moieties are connected by fumarate anions in a bis-monodentate mode. These 1-D chains are further connected by hydrogen bonds, generating a 2-D network. The variable temperature magnetic susceptibility measurement reveals that the magnetic couplings between Co(II) and NIT4Py are weak antiferromagnetic interactions.

Keywords: Crystal structure; Nitronyl nitroxide radical; Fumaric acid; Magnetic property

1. Introduction

For molecule-based magnets, metal–nitronyl nitroxide radical approach has attracted considerable attention [1, 2], and were used as bridging ligands, while strong electron-withdrawing coligands, such as hfac, pfbz, and pfpr were used as coligands. The steric hindrance of these coligands restricts the dimensionality of the metal–radical compounds. High-spin polynitroxides, which can bind to several metal centers and afford 3-D extended networks, were efficient and several high T_C molecule-based magnets were reported [3–14]. Another important strategy relies on functionalized nitronyl nitroxide radicals with electron-donating substituents, such as pyridine, imidazole, or benzimidazole. Based on their multidentate and strong electron-donor nature, many multi-dimensional magnetic materials have been obtained from this kind of radical ligand [15–17].

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Appropriate bridges, such as pseudohalido, dicyanido, terephthalato, and isophthalato anions, have also been used in designing and preparing multi-dimensional materials exhibiting spontaneous magnetic properties [18–22]. Fumarate is a good building block bridging based on its two carboxyl groups. Further, it can also act as hydrogen-bond acceptors and/or donors to generate supramolecular topologies [23, 24]. Taking advantage of the coordination abilities of both pyridyl-substituted nitronyl nitroxides and fumarate, a new metal–radical complex $\{[Co_2(NIT4Py)_4(fum)_2(H_2O)_4]\cdot(H_2O)_2\}_n$ is reported and its magnetic properties are studied.

2. Experimental

2.1. *Physical measurements*

Elemental analyses for C, H, N were carried out on a Model 1112 Flash EA elemental analyzer. The infrared (IR) spectrum was taken on a FT-IR 1730 spectrometer from 4000 to 400 cm^{-1} with KBr pellets. Variable temperature susceptibility measurements were carried out with a SQUID MPMS XL7 magnetometer from 2.0 to 300 K at a magnetic field of 2000 G. The molar susceptibility was corrected from the sample holder and diamagnetic contributions of all constituent atoms using Pascal's constants.

2.2. *Synthesis*

All reagents were A.R. grade and used without purification. The NIT4Py was prepared by the method described [25, 26]. Aqueous solution (5 mL) of dipotassium fumarate (0.05 mmol) was added to methanol solution of $Co(ClO_4)_2\cdot 6H_2O$ (0.05 mmol) and NIT4Py (0.1 mmol) at room temperature. The mixture was stirred for 2 h and then filtered. Deep blue-green single crystals suitable for X-ray structure analysis were grown at room temperature by slow evaporation of the filtrate. Anal. Calcd for $C_{56}H_{80}N_{12}O_{22}Co_2$ (%): C, 48.35; H, 5.796; N, 12.08. Found (%): C, 48.79; H, 6.159; N, 12.55. IR(KBr, cm^{-1}): $\nu_{asym}(COO^-)$, 1609 cm^{-1}; $\nu_{sym}(COO^-)$, 1400 cm^{-1}; $\nu(NO)$ of NIT4Py, 1375 cm^{-1}.

2.3. *X-ray crystallography*

A deep blue-green crystal of the title complex with approximate dimensions of 0.12 mm × 0.08 mm × 0.06 mm was put on a Bruker SMART APEXII CCD area detector equipped with graphite-monochromated Mo-Kα radiation ($\lambda = 0.71073$ Å). Data were collected at room temperature by φ–ω scan mode. The unit-cell parameters were determined from $1.62^\circ \le \theta \le 25.05^\circ$ for the complex. The details of data collection, crystallographic data, and structure refinement parameters are summarized in table 1. The structure was solved with direct methods using SHELXS-97 [27]. Hydrogens were assigned with common isotropic displacement factors and included in the final refinement by the use of geometrical restraints. A full-matrix least-squares refinement on F^2 was carried out using SHELXL-97 [28]. Reliability factors were defined as

Table 1. Crystallographic data for $\{[Co_2(NIT4Py)_4(fum)_2(H_2O)_4]\cdot(H_2O)_2\}_n$.

Empirical formula	$C_{56}H_{80}Co_2N_{12}O_{22}$
Formula weight	1391.18
Temperature (K)	273(2)
Wavelength (Å)	0.71073
Crystal system, space group	Triclinic, $P\bar{1}$
Unit cell dimensions (Å, °)	
a	9.8853(9)
b	13.2167(13)
c	13.8481(12)
α	68.5400(10)
β	73.467(2)
γ	74.596(2)
Volume ($Å^3$), Z	1587.6(3), 1
Calculated density ($Mg\,m^{-3}$)	1.455
Absorption coefficient (mm^{-1})	0.609
$F(000)$	730
θ range for data collection (°)	1.62–25.05
Limiting indices	$-10 \le h \le 11$, $-15 \le k \le 15$, $-16 \le l \le 15$
Reflections collected/unique	8337/5587 ($R_{int} = 0.0241$)
Observed reflections ($I > 2\sigma(I)$)	3311
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	5587/0/418
Goodness-of-fit on F^2	1.047
Final R indices ($I > 2\sigma(I)$)	$R_1 = 0.0662$, $wR_2 = 0.1780$
R indices (all data)	$R_1 = 0.1061$, $wR_2 = 0.2113$
Largest difference peak and hole ($e Å^{-3}$)	1.455 and −0.447
Weighing scheme ($P = (F_o^2 + 2F_c^2)/3$)	$w = 1/[\sigma^2(F_o^2) + (0.1195P)^2 + 0.7661P]$
$(\Delta/\sigma)_{max}$	0.003

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

Co(1)–O(3)	2.079(3)	Co(1)–O(5)	2.132(3)
Co(1)–N(3)	2.188(4)	O(1)–N(1)	1.276(5)
O(2)–N(2)	1.269(5)	O(3)–C(13)	1.222(6)
O(4)–C(13)	1.286(6)	Co(2)–O(8)	2.097(3)
Co(2)–O(10)	2.098(3)	Co(2)–N(6)	2.213(4)
O(6)–N(4)	1.273(5)	O(7)–N(5)	1.273(5)
O(8)–C(27)	1.205(7)	O(9)–C(27)	1.307(7)
O(3)–Co(1)–O(3)#1	180.0	O(5)–Co(1)–O(5)#1	180.0
N(3)#1–Co(1)–N(3)	180.0	O(3)–Co(1)–O(5)	89.8(1)
O(3)–Co(1)–N(3)	91.1(1)	O(3)–Co(1)–O(5)#1	90.2(1)
O(3)–Co(1)–N(3)#1	88.9(1)	O(5)–Co(1)–N(3)#1	87.6(1)
O(5)–Co(1)–N(3)	92.4(1)	O(8)#2–Co(2)–O(8)	180.0(2)
O(10)#2–Co(2)–O(10)	180.0	N(6)#2–Co(2)–N(6)	180.0
O(8)–Co(2)–O(10)	92.3(1)	O(8)–Co(2)–O(10)#2	87.7(1)
O(8)–Co(2)–N(6)	89.9(1)	O(8)–Co(2)–N(6)#2	90.1(1)
O(10)–Co(2)–N(6)	88.3(1)	O(10)–Co(2)–N(6)#2	91.7(1)

Symmetry transformations used to generate equivalent atoms: #1: $-x+1$, $-y+2$, $-z+1$; #2: x, $-y+1$, $-z+1$.

$R_1 = \Sigma(||F_o| - |F_c||)/\Sigma|F_o|$, and the function minimized was $wR_2 = (\Sigma w(|F_o|^2 - |F_c|^2)^2/\Sigma w|F_o|^2)^{1/2}$, where the unit weight was used in the least squares calculation. All non-hydrogen atoms were refined anisotropically. Selected bond distances and angles are listed in table 2.

3. Results and discussion

3.1. *Crystal structure*

$Co_2(NIT4Py)_4(fum)_2(H_2O)_4] \cdot (H_2O)_2$ consists of two similar types of neutral 1-D chains (Co1 chain and Co2 chain are shown in figure 1) and free water. In both chains, $[Co(NIT4Py)_2(H_2O)_2]$ moieties are connected by fumarate anions in a bis-monodentate mode. Each Co(II) is six-coordinate from two fumarate anions, two water molecules, and two NIT4Py ligands as *trans*-isomer. The central Co(II) is located at an inversion center in a distorted octahedral polyhedron. Bond angles in the octahedron are slightly distorted from the ideal values [87.6(1)–92.4(1)°].

In the Co1 chains, the bond lengths $Co1–O5_{water}$, $Co1–O3_{fum}$, and $Co1–N3_{(NIT4Py)}$ are 2.132(3), 2.079(3), and 2.188(4) Å, respectively. The dihedral angle between O2–N2–C3–N1–O1 and pyridyl ring in NIT4Py is 26.1°. In the Co2 chains, the bond lengths $Co2–O10_{water}$, $Co2–O8_{fum}$, and $Co2\text{-}N6_{(NIT4Py)}$ are 2.098(3), 2.097(3), and 2.213(4) Å, respectively. The dihedral angle between O7–N5–C17–N4–O6 and pyridyl ring of NIT4Py is 28.2°. The slight difference between Co1 chain and Co2 chain is observed from these structural parameters. Within the 1-D chains, the distance between successive Co's is 9.885 Å, corresponding to the cell length (*a*). In the title complex, the Co1 chains and the Co2 chains are alternatively arranged along the *ab* plane, generating an infinite 2-D network (figure 2) through interchain hydrogen bonds between fumarate anions and coordinated water $[O5 \cdots O9(-x+1, -y+1, -z+1) = 2.908\,Å]$. Hydrogen bonds exist between crystalline water and the Co2 chains $[O11 \cdots O10 = 2.634\,Å, O11 \cdots O8 = 2.772\,Å]$. The shortest interchain $Co1 \cdots Co2$ separation is 7.124 Å, and the $Co1 \cdots Co1$ (or $Co2 \cdots Co2$) separation is 13.217 Å, corresponding to cell length (*b*). The shortest interchain distance between N–O groups of NIT4Py is 3.457 Å. To the best of our knowledge, the title complex is the first example of nitronyl nitroxide radical complex with fumarate anion bridges.

3.2. *Magnetic property*

The plots of $\chi_M T$ and $1/\chi_M$ *versus* T for the title complex are shown in figure 3. Considering the structural characteristics, two independent $[Co(NIT4Py)_2]$ moieties are well-isolated and similar, so the magnetic data were interpreted considering only the isolated $[Co(NIT4Py)_2(fum)(H_2O)_2]$ units. The χ_M is the magnetic susceptibility per $[Co(NIT4Py)_2(fum)(H_2O)_2]$ unit.

At room temperature, $\chi_M T$ of the complex is $3.50\,cm^3\,K\,mol^{-1}$, which is much higher than expected for independent spins ($2.63\,cm^3\,K\,mol^{-1}$ for one $S_{Co} = 3/2$ spin and two $S_{Rad} = 1/2$ with $g = 2$), indicating that an important orbital contribution of Co(II) ($^4T_{1g}$) is involved. When the temperature is lowered, the $\chi_M T$ value decreases slowly, which indicates that exchange coupling between Co(II) and NIT4Py radical is weak antiferromagnetic. From the plot of $\chi_M{}^{-1}$ against T, the complex follows the Curie–Weiss law in the range 10–300 K, with a Curie constant of $3.66\,cm^3\,K\,mol^{-1}$. The Weiss temperature is $\theta = -13.60\,K$.

Based on the structure of the complex, the decrease in its $\chi_M T$ values with the lowering of temperature presumably originates from the following factors: (i) orbital angular momentum of the Co(II) center, (ii) magnetic interaction between Co(II) and

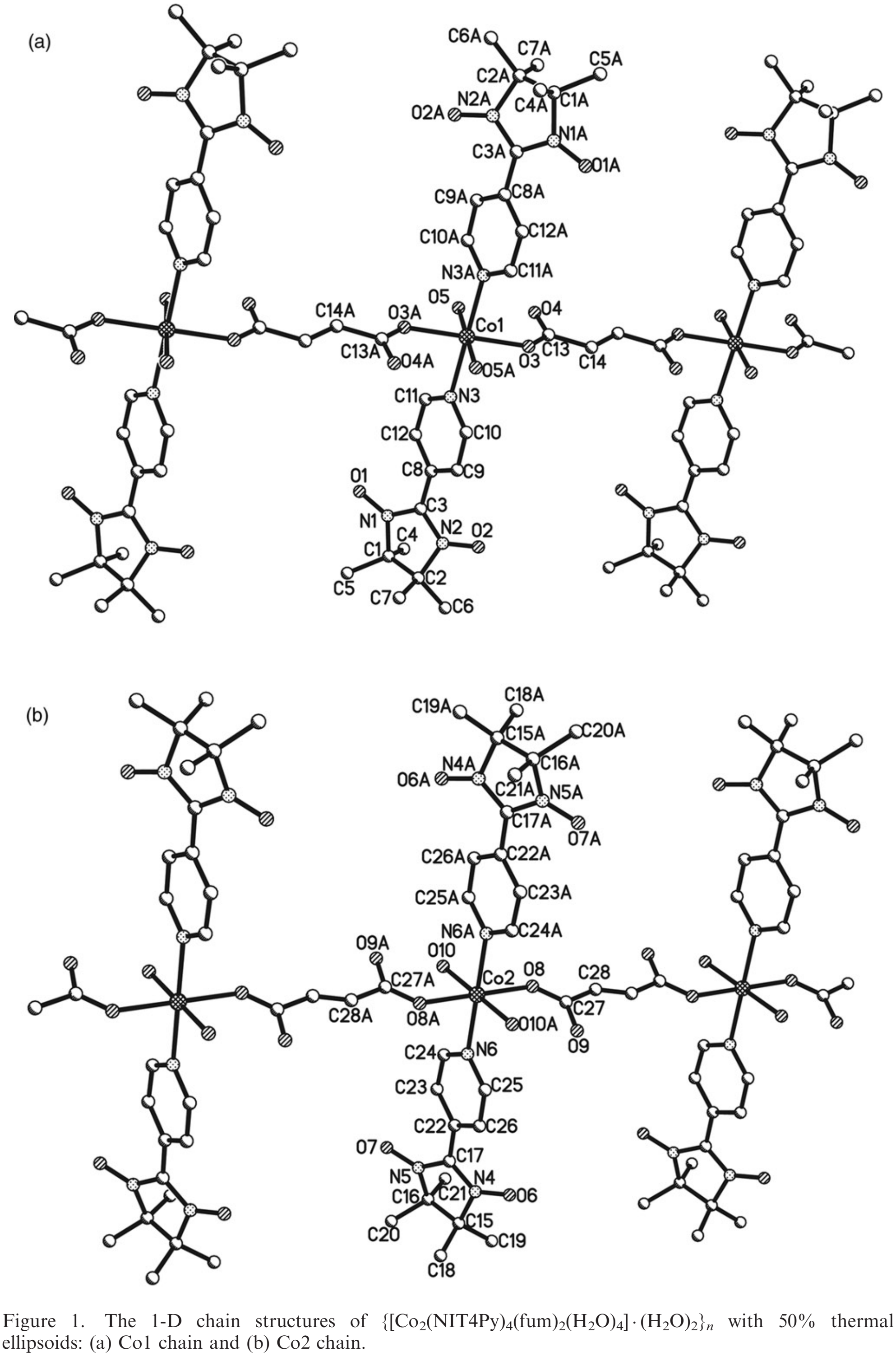

Figure 1. The 1-D chain structures of $\{[Co_2(NIT4Py)_4(fum)_2(H_2O)_4]\cdot(H_2O)_2\}_n$ with 50% thermal ellipsoids: (a) Co1 chain and (b) Co2 chain.

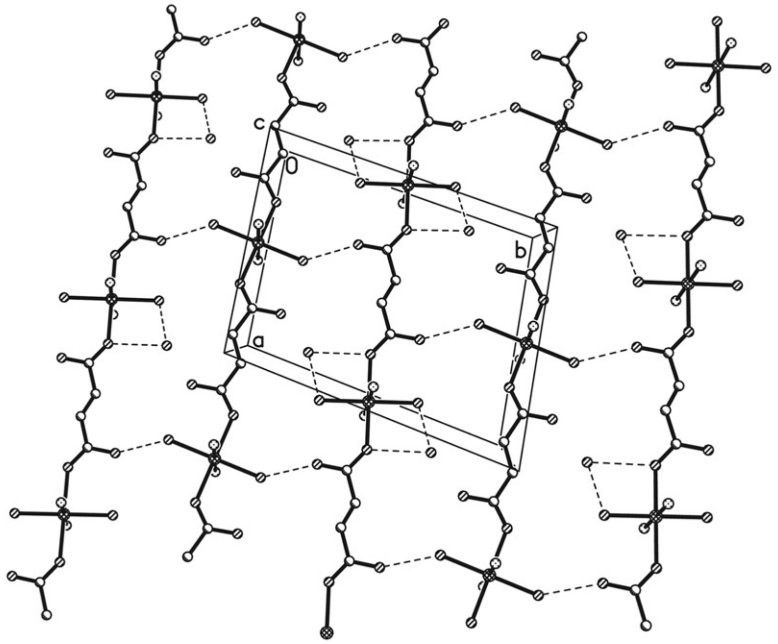

Figure 2. The 2-D network of $\{[Co_2(NIT4Py)_4(fum)_2(H_2O)_4]\cdot(H_2O)_2\}_n$ along the *ab* plane. For the sake of clarity NIT4Py groups have been omitted, except pyridyl-N.

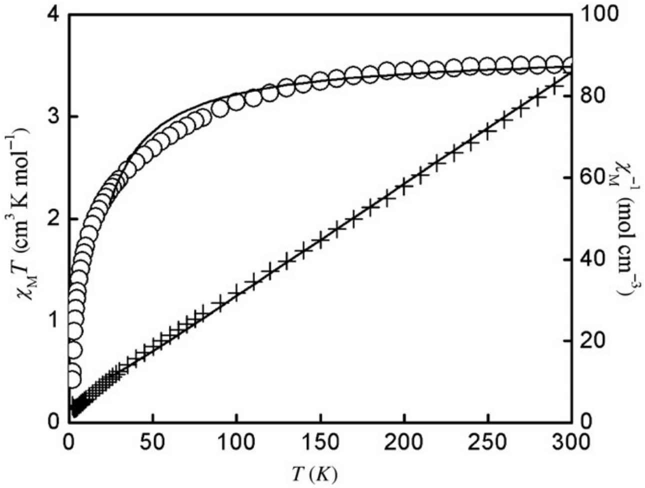

Figure 3. Plots of $\chi_M T$ (○) and χ_M^{-1} (+) *versus* T for the title complex. The solid line corresponds to the best theoretical fits.

coordinated NIT4Py radical through the pyridine ring, (iii) magnetic interaction between Co(II)–radical spin pairs through the fumarate anion bridge, and (iv) exchange coupling between neighboring nitroxide groups of NIT4Py radicals through space. A quantitative treatment of the susceptibility, including the orbital degeneracy of the

Co(II) center, cannot be carried out due to the lack of a theoretical model for the system. If the Co(II)–Co(II) exchange through fumarate bridge and the intrachain and interchain interactions were neglected, the simplified three-spin model Rad–Co(II)–Rad was used in the present system as a preliminary treatment. With the Hamiltonian $\hat{H} = -2J(\hat{S}_{\mathrm{Co}}\hat{S}_{\mathrm{rad1}} + \hat{S}_{\mathrm{Co}}\hat{S}_{\mathrm{rad2}})$, the magnetic susceptibility expression is given by Equation (1) with exchange coupling constant ($2J$) between Co(II) and the NIT4Py ligand.

$$\begin{aligned}\chi_{\mathrm{M}} &= \frac{Ng^2\beta^2}{4KT}\frac{A}{B} + N\alpha,\\ A &= 10 + \exp(-5\,\mathrm{J\,K^{-1}\,T^{-1}}) + 10\exp(-2\,\mathrm{J\,K^{-1}\,T^{-1}}) + 35\exp(3\,\mathrm{J\,K^{-1}\,T^{-1}}),\\ B &= 2 + \exp(-5\,\mathrm{J\,K^{-1}\,T^{-1}}) + 2\exp(-2\,\mathrm{J\,K^{-1}\,T^{-1}}) + 3\exp(3\,\mathrm{J\,K^{-1}\,T^{-1}}),\\ N\alpha &= 120 \times 10^{-6}\,\mathrm{cm^3\,mol^{-1}}.\end{aligned} \tag{1}$$

The least-squares fitting of magnetic susceptibility data in the temperature range from 300 to 24 K led to $g_{\mathrm{Co}} = 2.33$, $J = -4.91\,\mathrm{cm^{-1}}$, and $R = \Sigma(\chi_{\mathrm{obs}} - \chi_{\mathrm{calc}})^2/\Sigma(\chi_{\mathrm{obs}})^2 = 1.62 \times 10^{-3}$. The fitting below 24 K was unsuccessful, which may be attributed to spin–orbit coupling effect in Co(II). The fitting results show that the exchange coupling between the Co(II) and NIT4Py radical is weak anti-ferromagnetic interaction.

The magnetic coupling between Co(II) ion and NIT4py radical is dependent on the value of the dihedral angle between the pyridyl ring and the nitroxide group to some extent; the larger the value of the dihedral angle, the weaker the magnetic coupling. This can be explained by McConnell's mechanism [29]. According to this model, a spin distribution arising from the intramolecular spin polarization of the adjacent atoms leads to alternating positive and negative spin density on the carbon backbone of the radical ligands and delocalizes on the pyridyl ring. The positive spin density on the NO site of the radical induces negative spin density on the neighboring carbon due to spin polarization, which then induces negative spin density on the pyridyl nitrogen of NIT4Py radical. In the title complex, the dihedral angles between nitroxide moieties and the plane of pyridyl rings in NIT4Py were 26.1° or 28.2°, respectively. These large dihedral angles reduce the delocalization of the spin density of the NO groups, so exchange coupling between Co(II) and NIT4Py is weak ($-4.91\,\mathrm{cm^{-1}}$). This negative J value is comparable with the reported J values between Co(II) ion and NIT4Py: $-6.04\,\mathrm{cm^{-1}}$ for $[\mathrm{CoAu_2(CN)_4(NIT4Py)_2(H_2O)_2}]_n$ and $-4.53\,\mathrm{cm^{-1}}$ for $[\mathrm{Co(N(CN)_2)_2(NIT4Py)_2(H_2O)_2}]_n$, respectively [30, 31].

4. Conclusion

A 1-D Co(II)-radical chain complex $\{[\mathrm{Co_2(NIT4Py)_4(fum)_2(H_2O)_4}]\cdot(\mathrm{H_2O})_2\}_n$ was synthesized, in which fumarate was connected to successive Co(II) ions as μ-bridging ligands. There exist two types of 1-D chains (Co1 chain and Co2 chain), which are alternatively arranged along the *ab* plane. These 1-D chains are further linked by hydrogen bonds, generating an infinite 2-D network. Due to weak delocalization of the spin density of the NO groups in NIT4Py radical, the title complex has weak antiferromagnetic exchange couplings between Co(II) and NIT4Py.

Supplementary material

CCDC-759998 contains the 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, 12 Union Road, Cambridge CB2 1EZ, UK; Fax: +44 1223 336033).

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