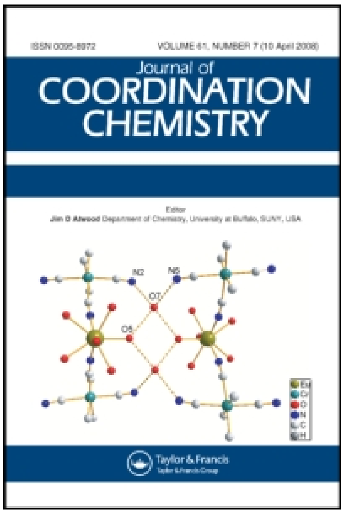

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Syntheses, structures, and magnetic properties of two 1-D dicyanamide manganese(III) complexes with Schiff-base ligands

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Syntheses, structures, and magnetic properties of two 1-D dicyanamide manganese(III) complexes with Schiff-base ligands

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Two new 1-D manganese(III) Schiff-base complexes bridged by dicyanamide (dca), $[Mn(III)(5\text{-}Brsalen)(dca)] \cdot CH_3OH$ (**1**) and $[Mn(III)(3,5\text{-}Brsalen)(dca)] \cdot CH_3OH \cdot CH_3CN$ (**2**) (5-Brsalen = *N*,*N*′-ethylenebis(5-bromo salicylaldiminato) dianion; 3,5-Brsalen = *N*,*N*′-ethylenebis(3,5-dibromosalicylal diminato) dianion), have been synthesized and characterized. X-ray diffraction analyses reveal that the two complexes have 1-D chain structures constructed by $\mu_{1,5}$-dca bridge. Magnetic susceptibility measurements exhibit weak antiferromagnetic exchange coupling in the complexes.

Keywords: 1-D Coordination polymer; Manganese(III); Schiff base; Dicyanamide; Magnetic properties

1. Introduction

Much research has focused on homoleptic and heteroleptic dicyanamide (dca)-bridged extended systems because of their fascinating structural and topological features and to understand the fundamentals of magnetic coupling and magneto-structure correlation [1–5]. The dca ligand, possessing three potential donor sites, coordinates to metal ions in various modes, terminal through a nitrile nitrogen [1], μ_2-1,3 bridge through the amido nitrogen and one nitrile nitrogen, end-to-end μ_2-1,5 bridge through two nitrile nitrogens [2], a three-atom μ_3 bridge through all of the nitrogens [3], an unusual μ_4-1,1,3,5 bridge with one of the two nitrile nitrogens connected to two metal ions [4], and a μ_5-1,1,3,5,5 bridge with both nitriles connected to two metal centers [5]. In most dca-containing metal complexes, the ligand is end-to-end (μ_2-1,5) bridging which mediates weak magnetic interactions between paramagnetic metal ions [6]. Factors to enhance magnetic coupling through this bridge are poorly understood and remain subject of intensive research [7].

Six-coordinate manganese(III) Schiff-base complexes display interesting structural and electronic effects [8–10]; variation of in-plane chelating and axial sites often leads

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to a change in the spin state of the metal ions: high-spin, low-spin, or spin-crossover state [11–13]. Accordingly, introducing dca into these Mn(III) Schiff-base systems combines their interesting characteristics and results in the formation of Mn(III)–dca complexes along with a rich coordination chemistry. Although many polymers of manganese with Schiff base have been reported [14–16], relatively a few Mn(III)–Schiff-base polymers have been found. In the present work, *N*,*N*′-ethylenebis(5-bromosalicylaldiminato) dianion (5-Brsalen) and *N*,*N*′-ethylenebis(3,5-dibromosalicylaldiminato) dianion (3,5-Brsalen) were chosen as Schiff bases and two 1-D dca manganese(III) complexes were synthesized and magnetostructurally characterized.

2. Experimental

2.1. *Materials and instrumentation*

All chemical reagents and organic solvents employed were of reagent grade quality and used without purification. H_2-5-Brsalen and H_2-3,5-Brsalen were synthesized by mixing the corresponding derivatives of salicylaldehyde and 1,2-diaminoethane in a 2 : 1 mole ratio in ethanol [17].

The elemental analyses for carbon, hydrogen, and nitrogen were carried out on a Model 240 Perkin-Elmer instrument. Diffraction data were collected at 294 K with a Bruker SMART 1000 CCD diffractometer. IR spectra were measured using KBr disks in a Bruker Tensor 27 FTIR spectrophotometer from 400 to 4000 cm^{-1}. Magnetic susceptibility data were recorded using a Quantum Design MPMS–7 SQUID Magnetometer from 2 to 300 K at an applied magnetic field of 1 KG for **1** and 5 KG for **2**.

2.2. *Preparation of the complexes*

2.2.1. Synthesis of [Mn(III)(5-Brsalen)(dca)] • CH_3OH (1). A solution of $Mn(ClO_4)_2 \cdot 6H_2O$ (0.0362 g, 0.1 mmol) in MeCN (10 mL) was added into a solution of H_2-5-Brsalen (0.0323 g, 0.1 mmol) in MeCN (10 mL), followed by the addition of NaOH (0.0080 g, 0.2 mmol). The dark-brown mixture was stirred for 30 min before the solution of dca (0.0089 g, 0.1 mmol) in CH_3OH (10 mL) was added. The resulting solution was continuously stirred for 1h and then filtered to get rid of the insoluble particles. Dark-brown crystals suitable for X-ray crystallographic analysis were obtained by slow evaporation of the filtrate in the course of a few weeks. The product was collected by filtration and washed with cool methanol and air dried. Yield: 55%. Anal. Calcd for ($C_{19}H_{16}Br_2MnN_5O_3$) (%): C, 39.54; H, 2.79; N, 12.14. Found (%): C, 39.10; H, 2.71; N, 12.50.

2.2.2. Synthesis of [Mn(III)(3,5-Brsalen)(dca)] • $CH_3OH \cdot CH_3CN$ (2). Complex **2** was prepared in an analogous manner to that of **1** but by using H_2-3,5-Brsalen (0.0410 g, 0.1 mmol) instead of H_2-5-Brsalen. Yield: 60%. Anal. Calcd for ($C_{21}H_{17}Br_4MnN_6O_3$) (%): C, 32.51; H, 2.21; N, 10.83. Found (%): C, 32.69; H, 1.85; N, 10.23.

2.3. *X-ray crystallography*

Diffraction data for **1** and **2** were collected at 294 K with a Bruker SMART 1000 CCD diffractometer using Mo-Kα radiation ($\lambda = 0.71073$ Å) with the ω–2θ scan technique. An empirical absorption correction (SADABS) was applied to raw intensities [18]. The structure was determined by direct methods (SHELX-97) and refined by full-matrix least-squares on F^2 using SHELX-97 [19]. The hydrogens were added theoretically, riding on the concerned atoms and refined with fixed thermal factors. Detailed crystallographic data for the two complexes are listed in table 1 and selected bond lengths and angles with their estimated standard deviations are listed in tables 2 and 3.

3. Results and discussion

3.1. *Crystal structures*

3.1.1. Structure of [Mn(III)(5-Brsalen)(dca)]•CH_3OH (1). Complex **1** crystallizes in the monoclinic, space group *P2₁/c* and possesses a 1-D zig-zag chain structure constructed by square-planar Mn(III)-5-Brsalen species bridged by dca ligands in an anti-anti configuration, as shown in figure 1. The Mn(III) ion is distorted octahedral coordinated by N_2O_2 donors of one 5-Brsalen dianion equatorial and two N donors from two different dca ligands in the axial positions. Each dca is a $\mu_{1,5}$ bridge linking monomeric $[Mn^{III}(5\text{-Brsalen})]^+$ into a 1-D zig-zag chain, in comparison to the NCNH-bridged Mn(III) chain [16]. The bond lengths of Mn(1)–O(1), Mn(1)–O(2), Mn(1)–N(1), and Mn(1)–N(2) are 1.881(3), 1.875(3), 1.988(4), and 1.988(4) Å, respectively, close to those in other Mn(III)–salen complexes [16, 20b], showing typical values for a high-spin Mn^{III} ion [20]. The N(3)–Mn(1)–N(5), Mn(1)–N(3)–C(17), Mn(1)–N(5)–C(18) angles in

Table 1. Crystal data and structure refinements for **1** and **2**.

Empirical formula	$C_{19}H_{16}Br_2MnN_5O_3$	$C_{21}H_{17}Br_4MnN_6O_3$
Formula weight	577.13	775.99
Crystal system	Monoclinic	Monoclinic
Space group	$P2_1/c$	$P2_1$
Crystal size (mm³)	0.20 × 0.18 × 0.12	0.20 × 0.18 × 0.14
Unit cell dimensions (Å, °)		
a	9.888(5)	7.565(3)
b	16.539(8)	16.299(6)
c	13.508(7)	10.999(4)
β	102.526(9)	96.842(6)
Volume (Å³)	2156.5(19)	1346.4(8)
Calculated density (mg m⁻³)	1.778	1.914
F (000)	1136	748
θ range for data collection (°)	1.98−26.48	1.86−25.02
Limiting indices	$-11 \le h \le 12$; $-20 \le k \le 15$; $-16 \le l \le 13$	$-8 \le h \le 8$; $-19 \le k \le 19$; $-8 \le l \le 13$
Data/parameters ratio	16.1	14.4
Goodness-of-fit on F^2	1.001	1.026
Final *R* indices [$I > 2\sigma(I)$]	$R_1 = 0.0459$, $wR_2 = 0.0954$	$R_1 = 0.0715$, $wR_2 = 0.1609$
R indices (all data)	$R_1 = 0.1139$, $wR_2 = 0.1202$	$R_1 = 0.1182$, $wR_2 = 0.1849$
Largest difference peak and hole (e Å⁻³)	0.689 and −0.588	0.859 and −0.765

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

Mn(1)–O(2)	1.875(3)	Mn(1)–N(3)	2.302(5)
Mn(1)–O(1)	1.881(3)	Mn(1)–N(5)	2.334(5)
Mn(1)–N(1)	1.988(4)	Mn(1)–N(2)	1.988(4)
O(2)–Mn(1)–O(1)	93.93(13)	N(2)–Mn(1)–N(5)	87.20(16)
O(2)–Mn(1)–N(1)	174.01(15)	N(3)–Mn(1)–N(5)	171.84(16)
O(1)–Mn(1)–N(1)	91.80(15)	C(1)–O(1)–Mn(1)	128.9(3)
O(2)–Mn(1)–N(2)	91.58(15)	C(16)–O(2)–Mn(1)	128.7(3)
O(1)–Mn(1)–N(2)	174.41(15)	C(7)–N(1)–C(8)	121.0(4)
N(1)–Mn(1)–N(2)	82.67(17)	C(7)–N(1)–Mn(1)	126.3(4)
O(2)–Mn(1)–N(3)	92.53(16)	C(8)–N(1)–Mn(1)	112.7(3)
O(1)–Mn(1)–N(3)	94.64(16)	C(10)–N(2)–Mn(1)	125.6(3)
N(1)–Mn(1)–N(3)	88.74(17)	C(9)–N(2)–Mn(1)	111.7(3)
N(2)–Mn(1)–N(3)	86.03(17)	C(17)–N(3)–Mn(1)	167.4(5)
O(2)–Mn(1)–N(5)	92.22(15)	C(18)–N(4)–C(17)	122.8(5)
O(1)–Mn(1)–N(5)	91.65(16)	C(18)–N(5)–Mn(1)	157.4(4)
N(1)–Mn(1)–N(5)	85.87(16)		

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

Mn(1)–O(2)	1.875(3)	Mn(1)–N(3)	2.302(5)
Mn(1)–O(1)	1.881(3)	Mn(1)–N(5)	2.334(5)
Mn(1)–N(1)	1.988(4)	Mn(1)–N(2)	1.988(4)
O(2)–Mn(1)–O(1)	93.93(13)	N(2)–Mn(1)–N(5)	87.20(16)
O(2)–Mn(1)–N(1)	174.01(15)	N(3)–Mn(1)–N(5)	171.84(16)
O(1)–Mn(1)–N(1)	91.80(15)	C(1)–O(1)–Mn(1)	128.9(3)
O(2)–Mn(1)–N(2)	91.58(15)	C(16)–O(2)–Mn(1)	128.7(3)
O(1)–Mn(1)–N(2)	174.41(15)	C(7)–N(1)–C(8)	121.0(4)
N(1)–Mn(1)–N(2)	82.67(17)	C(7)–N(1)–Mn(1)	126.3(4)
O(2)–Mn(1)–N(3)	92.53(16)	C(8)–N(1)–Mn(1)	112.7(3)
O(1)–Mn(1)–N(3)	94.64(16)	C(10)–N(2)–Mn(1)	125.6(3)
N(1)–Mn(1)–N(3)	88.74(17)	C(9)–N(2)–Mn(1)	111.7(3)
N(2)–Mn(1)–N(3)	86.03(17)	C(17)–N(3)–Mn(1)	167.4(5)
O(2)–Mn(1)–N(5)	92.22(15)	C(18)–N(4)–C(17)	122.8(5)
O(1)–Mn(1)–N(5)	91.65(16)	C(18)–N(5)–Mn(1)	157.4(4)
N(1)–Mn(1)–N(5)	85.87(16)		

1 are 171.84°, 166.59°, and 130.71°, respectively. The axial bond lengths of Mn(1)–N(3) and Mn(1)–N(5) (2.302(5) and 2.334(5) Å, respectively) are significantly longer than the corresponding values in salen–$\mathrm{Mn}^{\mathrm{III}}$ complexes {such as 2.25(3) Å in $[\mathrm{Mn(salen)CN}]_n$, 2.278(5) Å in {Mn[1,4-di(1-inidazolyl)butane](salen)}, 2.162(7) Å in [Mn(salen)(NCS)]} [11, 21, 22]. This axial elongation may be attributed to the Jahn–Teller distortion, which is commonly observed in octahedral $\mathrm{Mn}^{\mathrm{III}}$ complexes [23]. The two neighboring Mn(III) ions in the 1-D chain are separated with the shortest distances of Mn1 ⋯ Mn1A 8.436 Å.

3.1.2. Structure of [Mn(III)(3,5-Brsalen)(dca)] · CH_3OH · CH_3CN (2).

X-ray crystallography reveals that each manganese is six-coordinate, MnN_4O_2, in distorted octahedral geometry (figure 2). Two nitrogens and two oxygens of the 3,5-Brsalen

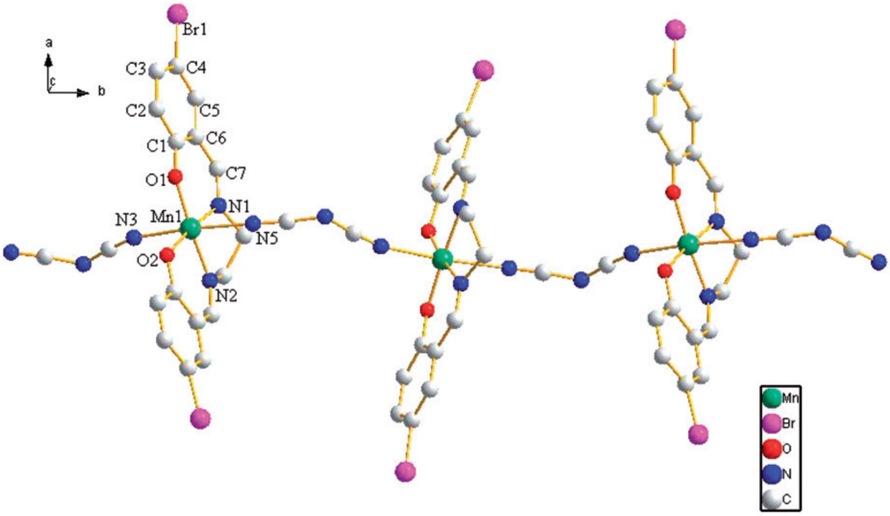

Figure 1. The 1-D chain structure in **1** (hydrogen atoms and solvent molecule have been omitted for clarity).

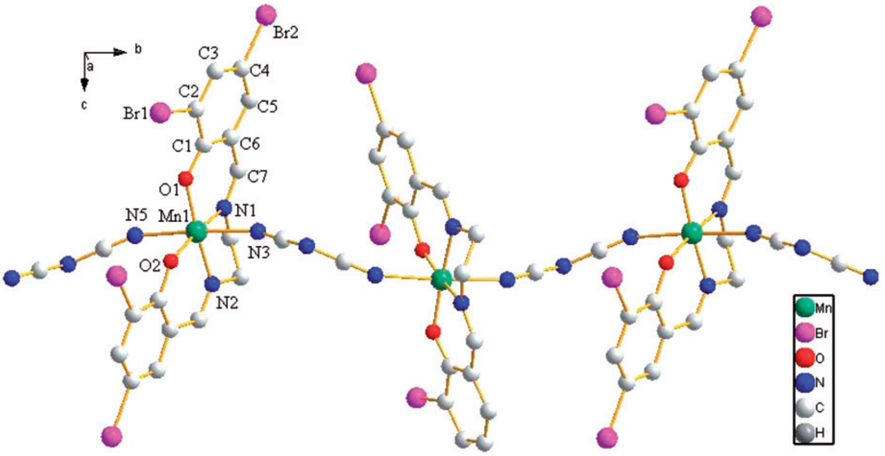

Figure 2. The 1-D chain structure in **2** (hydrogen atoms and solvent molecule have been omitted for clarity).

dianion define the equatorial plane around Mn(III), and the axial sites are occupied by two nitrogens of dca. The in-plane Mn–O and Mn–N bond lengths are 1.883(10), 1.888(8), 1.985(13), and 2.018(11) Å, respectively, similar to that in **1**. However, the average bond length Mn–N (2.330 Å) in the axial position is longer than that in **1** (2.318 Å). The N(3)–Mn(1)–N(5), Mn(1)–N(3)–C(17), and Mn(1)–N(5)–C(18) angles in **2** are 176.12°, 159.63°, and 150.89°, respectively. Two neighboring Mn(III) ions in the 1-D chain are separated with Mn1 $\cdots$ Mn1A distances of 8.383 Å. This is a unique 1-D one-stranded chain formed by Mn(III) and nitrogens (N3 and N5) of dca. Because adjacent crystal domains have opposite chirality (Flack parameter = 0.46(3)), the whole crystal is racemic and does not exhibit chirality.

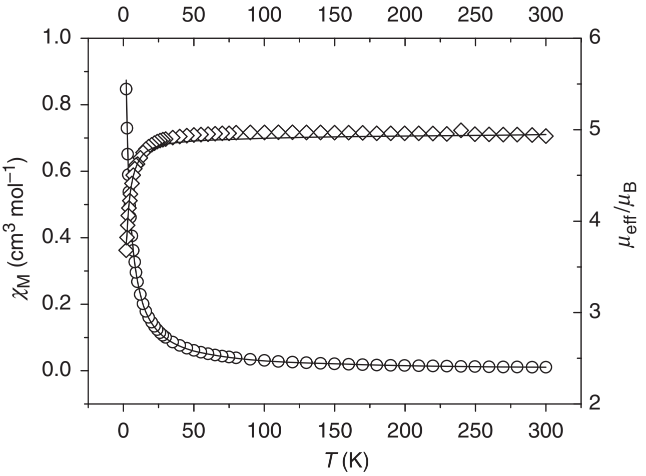

Figure 3. χ_m and μ_{eff} *vs.* T plots in an applied field of 1 KG with a theoretical fit for **1**.

3.2. *IR spectra*

The IR spectra of **1** and **2** exhibit very strong absorption bands at 2159, 2228, and 2289 cm^{-1} and 2157, 2220, and 2253 cm^{-1}, respectively, assigned to the vibrations of dca. The ν(C=N) stretching vibrations of the Schiff-base ligand appear at 1628 cm^{-1} for **1** and 1618 cm^{-1} for **2**. The ν_{as}(C–N) are at 1295 cm^{-1} for **1** and 1298 cm^{-1} for **2** and ν_s(C–N) at 905 cm^{-1} for **1** and 906 cm^{-1} for **2**. All these results are consistent with the crystal structures and are in good agreement with the reported literature [17, 24].

3.3. *Magnetic properties*

The temperature dependence of the magnetic susceptibility has been investigated at a field of 1 KG for **1** and 5 KG for **2** from 2 to 300 K. The plots of molar magnetic susceptibility χ_m *versus* temperature T and the effective magnetic moment μ_{eff} *versus* T for **1** and **2** are shown in figures 3 and 4. For **1**, the μ_{eff} value of 4.93 μ_B at room temperature is slightly larger than the spin-only value for high-spin Mn(III) (4.90 μ_B). On lowering the temperature, the μ_{eff} value decreases gradually from 300 to 25 K. When the temperature is lowered further, a sudden decrease of μ_{eff} occurs and reaches the minimum value of 3.68 μ_B at 2 K. For **2**, the μ_{eff} value of 4.90 μ_B at room temperature is in agreement with the spin-only value for high-spin Mn(III). On lowering the temperature, the μ_{eff} value decreases smoothly from 300 to 25 K, and then decreases abruptly from 25 to 2 K reaching the minimum value of 3.52 μ_B at 2 K.

Mn(III) usually has an important zero-field splitting [25]. Due to the single long $\mu_{1,5}$-dca bridge linking two neighboring Mn(III) ions, all exchange interactions in the structure must be very weak [26]. Thus, the decrease of $\chi_m T$ product in the low temperature region may be attributed to the zero-field splitting of the ground state of Mn(III) and/or very weak Mn(III)–Mn(III) magnetic interactions through the dca bridge. Thus, we analyzed the magnetic data of **1** and **2** by the following equations

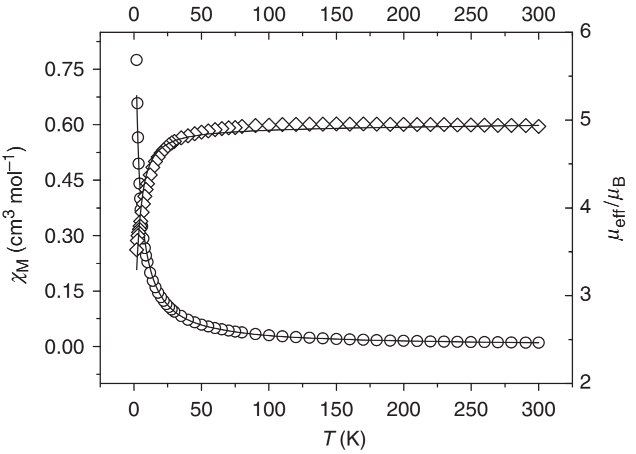

Figure 4. χ_m and μ_{eff} *vs.* T plots in an applied field of 5 KG with a theoretical fit for **2**.

including both single ion zero-field splitting (D) and weak magnetic coupling between Mn(III) ions with the use of molecular field approximation [27]:

$$\chi_{ZFC} = \frac{\chi_{\parallel} + 2\chi_{\perp}}{3}$$

$$\chi_{\parallel} = \frac{2Ng_{\parallel}^2\beta^2}{kT}\frac{\exp(-D/kT) + 4\exp(-4D/kT)}{1 + 2\exp(-D/kT) + 2\exp(-4D/kT)}$$

$$\chi_{\perp} = \frac{Ng_{\perp}^2\beta^2}{kT}\frac{(6/x)(1 - \exp(-x) + (4/3x)(\exp(-x) - \exp(-4x))}{1 + 2\exp(-x) + 2\exp(-4x)}$$

$$x = D/kT$$

$$\chi = \frac{\chi_{ZFC}}{1 - 2zJ\chi_{ZFC}/N\beta^2g^2}$$

The best agreement between the calculated and experimental values of the susceptibility was found with $g = 2.01$, $D = -2.94\,\text{cm}^{-1}$, $zJ' = -0.30\,\text{cm}^{-1}$, the agreement factor $R = 5.53 \times 10^{-4}$ for **1** and $g = 2.00$, $D = -3.78\,\text{cm}^{-1}$, $zJ' = -0.62\,\text{cm}^{-1}$, the agreement factor $R = 6.43 \times 10^{-3}$ for **2** (R is defined as $\Sigma(\chi_m^{calcd} - \chi_m^{bsd})^2/(\chi_m^{bsd})^2$). The D value is in good accordance with the reported literature [28, 29].

In conclusion, two new 1-D Mn(III) Schiff-base polymers bridged by dca were synthesized and characterized crystallographically and magnetically. X-ray diffraction analyses reveal that the two complexes have a 1-D chain structure constructed by $\mu_{1,5}$-dca bridge. The analysis of magnetic data shows that weak antiferromagnetic exchange couplings exist in the two complexes due to the long $\mu_{1,5}$-dca bridging ligand. Obviously, the ability to mediate the magnetic coupling of dca bridge is weaker than through the azido bridge [20a] or cyanamide ($NCNH_2$) bridge [16].

Supplementary material

Crystallographic data for the structural analyses have been deposited with the Cambridge Crystallographic Data Centre, CCDC Nos. 695500 **1**, and 695501

2, respectively. The copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge, CB21EZ, UK (Fax: +44 1223336 033; E-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

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