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# Synthesis, crystal structure and magnetic properties of a cyanide-bridged $Fe^{III}$–$Mn^{III}$ bimetallic chain based on $[Fe(bipy)(CN)_4]^-$ building block

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ABSTRACT

A cyano-bridged bimetallic chain complex of [Mn(salen)][Fe(bipy)(CN)$_4$] (**1**) (salen = *N*,*N*́-ethylenebis(salicylideneiminato)dianion; bipy = 2,2́-bipyridine), which was prepared by self-assembly of [Fe(bipy)(CN)$_4$]$^-$ and [Mn(salen)]$^+$ ions, has been characterized by elemental analyses, ICP, IR, thermoanalysis, single crystal X-ray structure analysis and magnetic measurements. In complex **1**, each [Fe(bipy)(CN)$_4$]$^-$ unit connects two [Mn(salen)]$^+$ units with its two *trans*-cyanide groups, and each [Mn(salen)]$^+$ unit is linked to two [Fe(bipy)(CN)$_4$]$^-$ ions in *trans*-positions, resulting in a straight one-dimensional neutral chain structure of complex **1**. The chains are stacking via mainly the aromatic π–π-type interactions. Magnetic studies reveal the presence of weak antiferromagnetic interactions between the adjacent $Fe^{III}$ and $Mn^{III}$ ions through cyanide-bridges.



## 1. Introduction

In the past decade, much effort has been devoted to the study of the magnetic interactions and magneto-structural correlations in molecular systems for the purpose of exploiting new molecule-based magnetic materials and interesting multifunctional molecule-based material such as single-molecule magnets (SMMs), single-chain magnets (SCMs), high critical temperature magnetic materials, spin-crossover materials, and multifunctional materials [1–4]. It has been found that cyanide-bridged bimetallic assemblies, based on $[M(CN)_6]^{n-}$ (M = Fe, Cr, Mn, Co) building blocks and hydrated metal ions $[M'(H_2O)_6]^{p+}$ or unsaturated coordinated complex $[M'(L)]^{m+}$, possess extraordinarily excellent magnetic performance and form a family of magnetic materials [5–7]. Recently, as an alternative strategy, the employment of capped molecular entities $[ML_p(CN)_q]^{n-}$ (L = polydentate ligand) instead of $[M(CN)_6]^{n-}$ as building blocks have been studied by several research groups [8–12]. The existence of the ancillary ligand (L) in the building blocks plays a key role in controlling the directions of structural propagation, and, as a result, generates low-dimensional magnetic assemblies. Nowadays, A few series of bimetallic assemblies in this new family with interesting magnetic properties such as SMM and SCM have been reported [13–16]. On the other hand, with enhanced Jahn–Teller effects and magnetic anisotropy, $Mn^{III}$–Schiff base complexes have been demonstrated as good precursors in constructing cyanide-bridged magnetic materials [17–21]. In our continuous efforts to assemble new cyanide-bridged magnetic complexes, we are now exploring the use of tailored cyanometalate complexes as building blocks. In the paper, we present a new cyanide-bridged chain complex [Mn(salen)][Fe(bipy)(CN)$_4$](**1**) obtained by assembly of a tailored cyanometalate complex of [Fe(bipy)(CN)$_4$]$^-$ and a $Mn^{III}$–Schiff base complex of [Mn(salen)]$^+$ (bipy = 2,2′-bipyridine; salen = *N*,*N*́-ethylenebis(salicylideneiminato)dianion)), and the magnetic and thermal properties of complex **1** were investigated.

## 2. Experimental

### 2.1. Physical measurements

Elemental analyses for C, H and N were performed at a Perkin-Elmer 240C microanalysis instrument (USA). Mn and Fe analyses were made on a Jarrell-Ash 1100+2000 inductively coupled plasma quantometer. IR spectra were recorded on a Nicolet FT-170SX spectrometer with KBr pellets in the 4000–400 cm$^{-1}$ region. The thermoanalysis was measured by a NETZSCH STA449C differential scanning calorimeter. The magnetic measurements were per-

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formed using a Quantum Design MPMS-XL SQUID magnetometer. Diamagnetic corrections were made using Pascal's constants. Effective magnetic moments were calculated using the equation $\mu_{eff} = 2.828(\chi_M \times T)^{1/2}$, where $\chi_M$ is the magnetic susceptibility per formula unit.

### 2.2. Preparations

All chemicals and solvents were reagent grade and were used without further purification. The precursors of $PPh_4[Fe^{III}(bipy)(CN)_4]\cdot H_2O$ [22] and $[Mn^{III}(salen)]ClO_4\cdot 2H_2O$ [20,23] were synthesized according to the methods reported previously.

#### 2.2.1. Caution

Perchlorate salts of metal complexes with organic ligands are potentially explosive and should be handled in small quantities with great care.

#### 2.2.2. [Mn(salen)][Fe(bipy)(CN)$_4$] (**1**)

A solution of $[Mn^{III}(salen)]ClO_4\cdot 2H_2O$ (0.1 mmol) in methanol (10 ml) was added to a solution of $PPh_4[Fe(bipy)(CN)_4]\cdot H_2O$ (0.1 mmol) in acetonitrile (10 ml). The resulting solution was filtrated and the filtrate was left to allow slow evaporation in dark at room temperature. Black crystals of complex **1** were obtained after two days, washed with MeOH and $H_2O$, respectively, and dried in air. Anal. Found: C, 56.31; H, 3.51; N, 17.44; Fe, 8.80; Mn, 8.53%. Calcd. for $C_{30}H_{22}FeMnN_8O_2$: C, 56.54; H, 3.48; N, 17.58; Fe, 8.76; Mn, 8.62%. IR: $\nu_{C\equiv N}$(cyanide): 2133, 2118 cm$^{-1}$.

### 2.3. X-ray crystallography

Diffraction data were collected at 291(2) K on a Bruker SMART APEX CCD area detector diffractometer using graphite-monochromated Mo-K$\alpha$ radiation ($\lambda = 0.71073$ Å) with the $\varphi$ and $\omega$ scan mode. Empirical absorption correction was made with SADABS. The structure was solved by direct methods and refined by full matrix least-squares techniques based on $F^2$. All non-hydrogen atoms were refined with anisotropic thermal parameters. The idealized positions of the hydrogen atoms were located by using a riding model. All computations were carried out using the SHELXTL-PC program package.

Crystal data for **1**: $C_{30}H_{22}FeMnN_8O_2$, $M = 637.35$, monoclinic, space group $P2(1)/c$ (No. 14), $a = 9.4749(7)$, $b = 21.2029(15)$, $c = 13.6263(10)$ Å, $\beta = 92.262(1)^\circ$, $U = 2735.3(3)$ Å$^3$, $Z = 4$, $D_c = 1.548$ g/cm$^3$, $F(000) = 1300$, $\mu = 1.037$ mm$^{-1}$ and $S = 1.01$. 14815 reflections measured, 5373 unique ($R_{int} = 0.037$). The final $R_1 = 0.0316$ and $wR_2 = 0.0830$ for 4129 observed reflections [$I > 2\sigma(I)$] and 379 parameters.

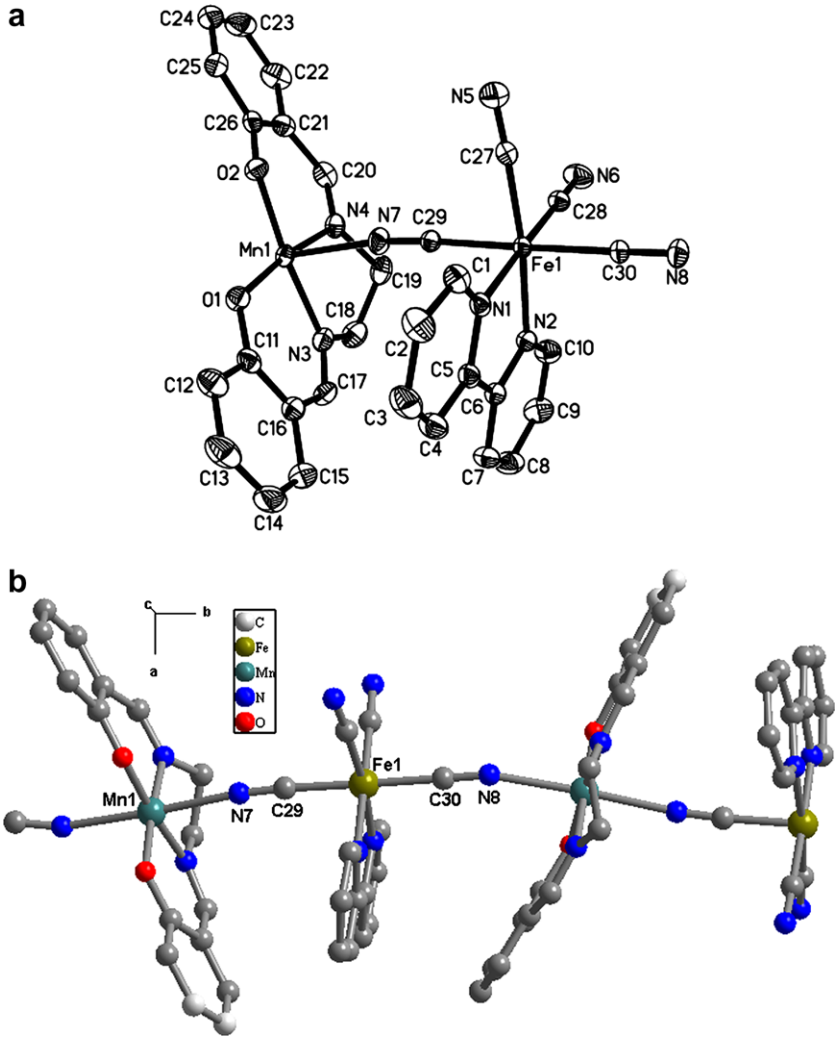


**Fig. 1.** Molecular view of complex **1** showing (a) the atom-labeling scheme and (b) bimetallic chain structure.

## 3. Results and discussion

### 3.1. Crystal structure

The structure of complex **1** is shown in Figs. 1 and 2. The selected bond distances and angles are listed in Table 1. The molecular structure of complex **1** consists of one $[Fe(bipy)(CN)_4]^-$ anion and one $[Mn^{III}(salen)]^+$ cation (Fig. 1a). Each $[Fe(bipy)(CN)_4]^-$ anion coordinates with two $[Mn^{III}(salen)]^+$ cations via two *trans*-$CN^-$ groups, whereas each $[Mn^{III}(salen)]^+$ cation is linked to two $[Fe(bipy)(CN)_4]^-$ ions in *trans*-conformation, which results in a straight neutral Fe–Mn bimetallic chain composed of $[Fe(bipy)(CN)_4]^-$ and $[Mn(salen)]^+$ alternately linked by the cyanide-bridges (Fig. 1b). As usual, the $[Fe(bipy)(CN)_4]^-$ fragment exhibits a distorted octahedral structure consisting of two N atoms from a planar bipy and four C atoms from four $CN^-$ groups. The Fe—C≡N angles for both terminal [176.4(2)° and 178.5(2)°] and bridging [176.5(2)° and 179.1(2)°] $CN^-$ are somewhat below that of strict linearity. The Fe–C distances for the bridging $CN^-$ [1.961(2) and 1.947(2) Å] are slightly longer than those for the terminal $CN^-$ [1.917(2) and 1.923(2) Å], while the C–N distances of the bridging $CN^-$ [1.140(3) and 1.131(3) Å] are almost equal to those of the terminal $CN^-$ [1.140(3) and 1.141(3) Å]. The values of the Fe–N(bipy) bond distances [1.986(2) and 1.987(2) Å] and that of the angle subtended by the chelating bipy [80.6(1)° at N(1)–Fe(1)–N(2)] are practically the same as those observed in the mononuclear $PPh_4[Fe(bipy)(CN)_4]$ and the trinuclear species $[\{Fe^{III}(bipy)(CN)_4\}_2M^{II}(H_2O)_4]\cdot 4H_2O$ (M = Mn or Zn) [22]. The $Mn^{III}$ ion assumes a distorted octahedral coordination geometry, in which the equatorial sites are occupied by $N_2O_2$ donor atoms of the quadridentate Schiff base ligand salen with the Mn–N/O bond distances of 1.880–1.992 Å, while the axial positions are occupied by two nitrogen atoms from bridging $CN^-$ groups with Mn–$N_{ax}$ distances of 2.282 and 2.329 Å. The axial elongation accounts for the existence of the well-known Jahn–Teller effect on an octahedral high-spin $Mn^{III}$ ion. The Mn—N≡C bond angles deviate significantly from linearity with the angles of Mn(1)–N(7)–C(29) = 171.3(2) and Mn(1B)–N(8)–C(30) = 159.6(2)°. In addition, the bonding parameters of the salen ligand are close to those found in related complexes reported previously [20–21].

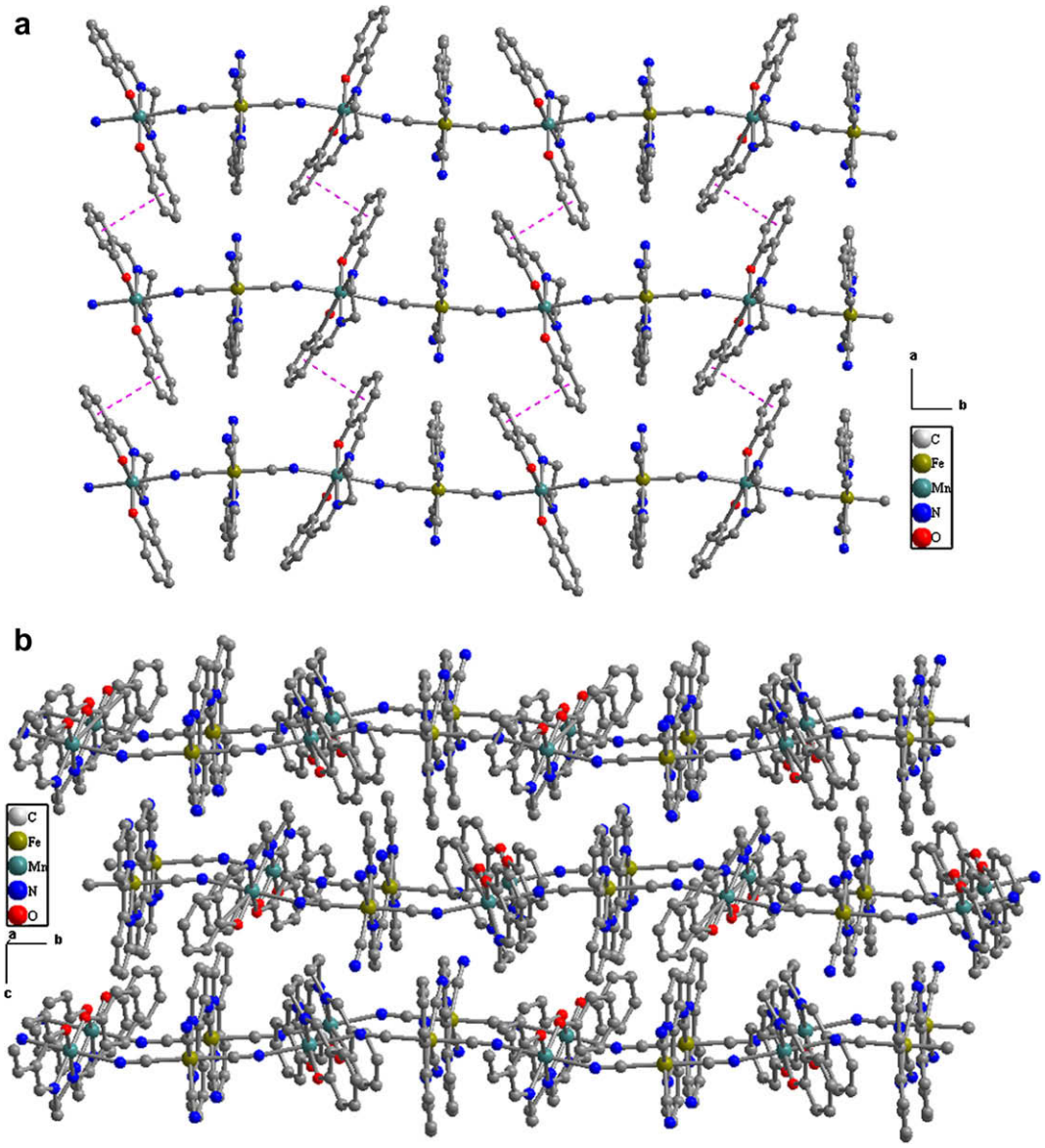


**Fig. 2.** (a) Projection of complex **1** along the *c*-axis, showing the layer structure. (b) Packing diagram viewed from the *a*-axis.

**Table 1**
Selected bond distances (Å) and bond angles (°) for complex **1**.

| | | | |
|---|---|---|---|
| *Bond distances* | | | |
| Fe1–N1 | 1.9865(18) | Mn1–O1 | 1.8947(14) |
| Fe1–N2 | 1.9868(18) | Mn1–O2 | 1.8801(14) |
| Fe1–C27 | 1.917(2) | Mn1–N3 | 1.9917(18) |
| Fe1–C28 | 1.923(2) | Mn1–N4 | 1.9803(18) |
| Fe1–C29 | 1.947(2) | Mn1–N7 | 2.2819(17) |
| Fe1–C30 | 1.961(2) | Mn1–N8A | 2.3292(17) |
| *Bond angles* | | | |
| N1–Fe1–N2 | 80.61(7) | N4–Mn1–N8A | 89.39(7) |
| N1–Fe1–C27 | 97.39(8) | N7–Mn1–N8A | 177.29(7) |
| N1–Fe1–C28 | 176.18(8) | O1–Mn1–N7 | 90.97(6) |
| N1–Fe1–C29 | 88.74(7) | Mn1B–N8–C30 | 159.62(17) |
| N1–Fe1–C30 | 91.33(7) | Mn1–N7–C29 | 171.28(17) |
| N2–Fe1–C27 | 173.53(7) | Fe1–C27–N5 | 176.41(19) |
| N2–Fe1–C28 | 95.68(8) | Fe1–C29–N7 | 176.54(18) |
| N2–Fe1–C29 | 86.29(7) | Fe1–N1–C1 | 126.11(16) |
| N2–Fe1–C30 | 94.82(7) | Fe1–N1–C5 | 115.39(14) |
| C27–Fe1–C28 | 86.20(9) | Fe1–N2–C6 | 115.50(14) |
| C27–Fe1–C29 | 87.52(8) | Fe1–N2–C10 | 125.18(16) |
| C27–Fe1–C30 | 91.37(8) | Fe1–C28–N6 | 178.54(19) |
| C28–Fe1–C29 | 90.10(8) | Fe1–C30–N8 | 179.09(18) |
| C28–Fe1–C30 | 89.90(8) | | |
| C29–Fe1–C30 | 178.89(8) | | |

Symmetry transformations used to generate equivalent atoms: $A = 1 - x, -1/2 + y, 3/2 - z$; $B = 1 - x, 1/2 + y, 3/2 - z$.

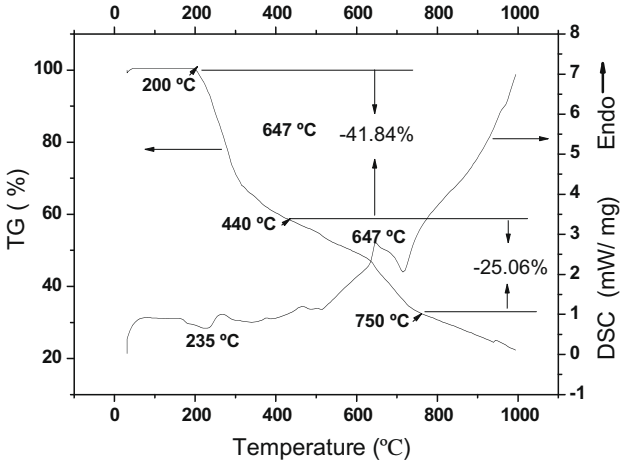


**Fig. 3.** TG-DSC curves of complex **1**.

The intrachain Fe···Mn distances through the $CN^-$ bridges are 5.336 and 5.351 Å, which is somewhat agreement with that of the Fe–Mn system with mpzcq and pzcq [24–25]. The nearest interchain Fe···Mn, Mn···Mn and Fe···Fe distances is 6.771, 6.513 and 7.288 Å. It is known that the packing of the bimetallic chains is often dominated by aromatic π–π stacking interactions in the case of the presence of large aromatic ligands [21]. The chains in complex **1** are running along the *b* direction, and form layers parallel to the *ab* plane. In the layer each chain interacts with two other adjacent chains via face-to-face π–π stacking between the benzene rings of the salen ligand of the $[Mn(salen)]^+$ units (Fig. 2a). The plane-to-plane distance is 3.81 Å, and the dihedral angle is 7°. The adjacent chains are also held together by the weak H-bonds between the terminal cyanides of $[Fe(bipy)(CN)_4]^-$ units in one chain and the C–H donors of bipy ligands in neighboring chains. The 3D structure is formed by the layers stacking along *c* direction mainly via van der Waals interaction (Fig. 2b).

### 3.2. Thermal analysis

The thermoanalysis was performed in nitrogen atmosphere with a heating rate of 10 °C $min^{-1}$. Fig. 3 shows the TG-DSC curve of complex **1**. With increasing temperature, the complex can keep stable until 200 °C. After that, the complex starts to decompose with a series of continuous weight loss processes. The weight loss of 41.84% from 200 to 440 °C is consistent with the loss of the ligand salen (calcd. 41.78%). The following weight loss of 25.06% from 440 to 750 °C might be due to the loss of the ligand bipy (calcd. 24.51%). When further heating, the decomposition continues with further weight loss.

### 3.3. Magnetic properties

The magnetic susceptibilities of complex **1** were measured with an applied field $H = 2$ kOe in the temperature range 1.8–300 K. The plots of $\chi_M T$ *vs. T* and $1/\chi_M$ *vs. T* are given in Fig. 4. At room temperature, the $\chi_M T$ per MnFe unit is 3.49 emu K $mol^{-1}$ (5.28 $\mu_B$), which is consistent with the spin-only value of 3.38 emu K $mol^{-1}$ (5.2 $\mu_B$) expected for an uncoupled spin system (one $S_{Fe} = 1/2$, one $S_{Mn} = 2$) with $g = 2.0$. On lowering the temperature, the $\chi_M T$ value almost keeps constant until 60 K, then gradually decreases to 3.0 emu K $mol^{-1}$ (4.9 $\mu_B$) at 14 K, finally sharply decreases with further decreasing temperature to 1.62 emu K $mol^{-1}$ (3.6 $\mu_B$) at 1.8 K. The plot of $1/\chi_M$ *vs. T* obeys the Curie–Weiss law with a negative Weiss constant $\theta = -2.6$ K. These results indicate the presence of a weak antiferromagnetic interaction in complex **1**, which can be attributed to antiferromagnetic coupling between $Fe^{III}$ and $Mn^{III}$ ions through cyanide-bridges. The field dependence of the magnetization measured at 1.8 K is shown in Fig. 5. The magnetization cannot reach saturation until 70 kOe. The experimental curve is always lower than that derived from the Brillouin function at 1.8 K, confirming further antiferromagnetic coupling between the adjacent $Fe^{III}$ and $Mn^{III}$. The antiferromagnetic interaction between the $Mn^{III}$ ($S = 2$) and $Fe^{III}$ ($S = 1/2$) can be rationalized in terms of the overlap of the magnetic orbitals of these ions [5]. It is interesting to note that the magnetism of complex **1** is obviously different from that of its $Cr^{III}$–$Mn^{III}$ analogs [21], which are metamagnets showing antiferromagnetic to ferrimagnetic transition due to strong intrachain uncompensated antiferromagnetic coupling and weak interchain antiferromagnetic coupling. In $Cr^{III}(t_{2g}^3 e_g^0)$–$Mn^{III}(t_{2g}^3 e_g^1)$ there are nine antiferromagnetic exchanging approaches via electrons in the $t_{2g}$(Cr) and $t_{2g}$ (Mn) orbitals, while in the situation of $Fe^{III}(t_{2g}^5 e_g^0)$–$Mn^{III}(t_{2g}^3 e_g^1)$ here there are only one-third the antiferromagnetic approaches as the $Cr^{III}$–$Mn^{III}$ pair has

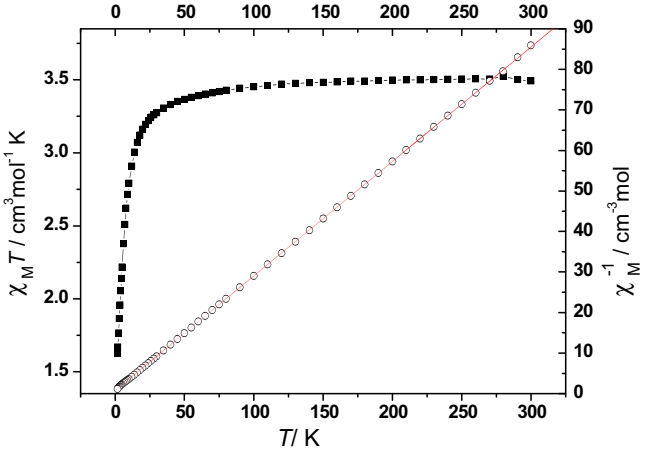


**Fig. 4.** Temperature dependence of $\chi_M T$ (■) and $1/\chi_M$ (○) for complex **1** measured at 2 kOe. The solid line represents the fit obtained by the Curie–Weiss law (see text).

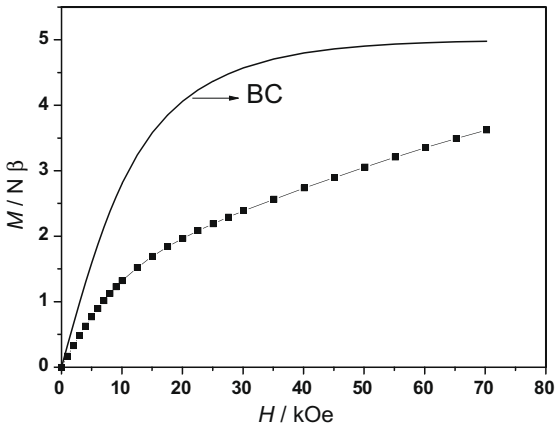


**Fig. 5.** Field dependence of the magnetization for **1** at 1.8 K. The Brillouin curve (BC) for the independent one $Fe^{III}$ and one $Mn^{III}$ ions is given at 1.8 K.

because there is only one unpaired electron in the $t_{2g}$ orbitals of $Fe^{III}$. Therefore, the $Fe^{III}$–$Mn^{III}$ antiferromagnetic coupling in complex **1** is much weaker than the $Cr^{III}$–$Mn^{III}$ antiferromagnetic coupling in the $Cr^{III}$–$Mn^{III}$ analogs and, as a result, unlike the $Cr^{III}$–$Mn^{III}$ analogs, complex **1** cannot form the ferrimagnetic chains. Of course, it is possible that there exists a very weak interchain antiferromagnetic interaction in complex **1**, which might contribute to the overall antiferromagnetic property of this complex [21]. Furthermore, complex **1** also supplies an additional example confirming that the substitution of $Cr^{III}$ with $Fe^{III}$ will result in a weaker magnetic coupling in cyanide-bridged bimetallic systems [5].

## 4. Conclusion

A new cyano-bridged bimetallic assembly, [Mn(salen)][Fe(bipy)$(CN)_4$], has been synthesized and characterized structurally and magnetically. This complex has a straight chain structure as the $[Fe(bipy)(CN)_4]^-$ unit, with its two *trans*-cyanide ligands, connects the two neighboring $[Mn(salen)]^+$ units at axial positions. The chains stack via mainly the aromatic π–π-type interactions. The complex shows a weak antiferromagnetic interaction between the adjacent $Fe^{III}$ and $Mn^{III}$ ions through cyanide-bridges. The present example further demonstrates $[Fe(bipy)(CN)_4]^-$ as an effective building block in constructing cyano-bridged low-dimensional systems. The synthetic approach can be extended to explore bimetallic systems with other transition metals, and this may bring about a deeper understanding of the magnetic interaction in molecule-based magnetic materials.

## 5. Supplementary materials

CCDC 709293 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via http://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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