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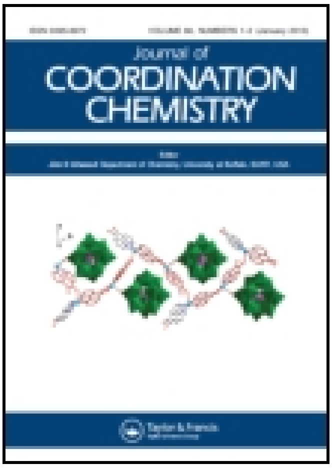

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An antiferromagnetic metal-organic framework with high symmetry octanuclear $Mn_8(\mu_4\text{-}O)_3(COO)_{12}$ secondary building units

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An antiferromagnetic metal-organic framework with high symmetry octanuclear $Mn_8(\mu_4\text{-}O)_3(COO)_{12}$ secondary building units

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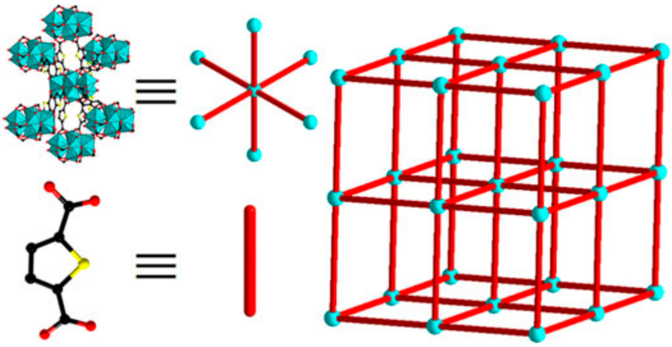

A new antiferromagnetic metal-organic framework, $\{[(CH_3)_2NH_2]_2[Mn_8(\mu_4\text{-}O)_3(thb)_6(solvent)_6]\}_n$ (thb = 2,5-thiophenedicarboxylate) with octanuclear $Mn_8(\mu_4\text{-}O)_3(COO)_{12}$ clusters as secondary building unit was synthesized and characterized, which has a *pcu* network topology when considering the Mn_8 cluster as a 6-connected node and each two double-linking thb ligands as a linker.

A new antiferromagnetic 3-D metal-organic framework $\{[(CH_3)_2NH_2]_2[Mn_8(\mu_4\text{-}O)_3(thb)_6(solvent)_6]\}_n$ (**1**, thb = 2,5-thiophenedicarboxylate) with octanuclear $Mn_8(\mu_4\text{-}O)_3(COO)_{12}$ secondary building unit (SBU) has been solvothermally synthesized and characterized by single-crystal X-ray diffraction, thermogravimetric analysis, and infrared spectroscopy. Considering this Mn_8 SBU as a 6-connected node, the overall framework of **1** has a *pcu* topology. It is interesting that the high symmetry Mn cluster shows a double-linked connection between two SBUs and exhibits an antiferromagnetic property.

Keywords: Metal-organic framework; Mn_8 cluster; *pcu* Topology; Antiferromagnetic property

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1. Introduction

As a new class of porous materials, metal-organic frameworks (MOFs) have attracted much attention not only for their fascinating structural architectures, but also for their potential applications in gas storage, separation, sensing, heterogeneous catalysis, fluorescence, and magnetism [1–18]. As low-density materials, there are many reports about the magnetic properties of MOFs for exploiting their applications in magnetic information storage, magnetic sensors, molecular switches, and magnetic refrigeration [13–15]. The architectures of MOFs play an important role in their magnetic properties. The metal centers (single metal ions or second building units (SBUs)) and organic ligands are also important factors for the synthesis of MOFs [19]. On the premise of selected organic ligand and metal salt, the formation of SBUs usually guides the structure of MOFs and dominates the properties of resulting MOFs. For example, MOF-5 has tetrameric $[Zn_4O(CO_2)_6]$ SBU; MIL-101 is based on trimeric $[M^{III}{}_3O(CO_2)_6]$ SBU; and some PCN-type frameworks are formed from dimeric $[Cu_2(CO_2)_4]$ SBU [20–22]. Until now, lots of Mn-based compounds with multinuclear Mn clusters acting as SBUs have been reported. However, MOFs containing high symmetric SBUs with more than eight metal ions are rare [23–26]. In this work, we have synthesized and characterized a 3-D MOF $\{[(CH_3)_2NH_2]_2[Mn_8(\mu_4\text{-}O)_3(thb)_6(solvent)_6]\}_n$ (**1**, thb = 2,5-thiophenedicarboxylate) with $Mn_8(\mu_4\text{-}O)_3(COO)_{12}$ SBU; the magnetic properties of the complex are also investigated.

2. Experimental

2.1. *Materials and general methods*

Commercially available reagents were used as received. 2,5-Thiophenedicarboxylic acid was purchased from Aldrich Chem. Co. FTIR data were recorded on an IRAffinity-1 instrument. Thermogravimetric analysis (TGA) data were obtained on a TGA-50 (SHIMADZU) thermogravimetric analyzer at a heating rate of 10 °C min^{-1} under N_2. Magnetic data were collected using crushed crystals of the sample on a Quantum Design PPMS-9T magnetometer. The data were corrected using Pascal's constants to calculate the diamagnetic susceptibility. In the measurement, the sample was wrapped by a film then fixed in the tube, and the background of the film was corrected.

2.2. *Synthesis of $\{[(CH_3)_2NH_2]_2[Mn_8(\mu_4\text{-}O)_3(thb)_6(solvent)_6]\}_n$ (1)*

1 was synthesized by a solvothermal reaction procedure: a DMF (1.5 mL) solution containing 2,5-thiophenedicarboxylic acid (H_2thb, 34.4 mg, 0.2 mM), $Mn(NO_3)_2 \cdot 4H_2O$ (50.2 mg, 0.2 mM), and five drops of HBF_4 (48% in water) were sealed in a Pyrex tube, heated at 100 °C for three days, and then cooled to room temperature. Colorless block crystals of **1** were collected, with a yield of 48% based on the Mn(II) salt.

2.3. *Crystal structure determination*

Single-crystal X-ray diffraction measurement was carried out on a Bruker Smart 1000 diffractometer equipped with graphite-monochromated Mo*Kα* radiation (λ = 0.71073 Å) with

Table 1. The crystallographic and experimental data for **1**.

Formula	$C_{36}H_{12}C_{10}Mn_8O_{33}S_6$
M	1604.34
Crystal system	Hexagonal
Space group	*R-3c*
a (Å)	15.243(1)
b (Å)	15.243(1)
c (Å)	56.173(4)
α (°)	90
β (°)	90
γ (°)	120
V (Å^3)	11,303(1)
Z	6
D_C (g cm^{-3})	1.414
μ (mm^{-1})	1.531
λ (Å)	0.71073
T (K)	296(2)
Reflections collected	27,252
Observed data	3830
$R_1(I > 2\delta)$	0.0597
$wR_2(I > 2\delta)$	0.1753

$R_1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$ and $wR_2 = \{\Sigma[w(F_o^2 - F_c^2)^2]/[w(F_o^2)^2]\}^{1/2}$, $[F_o > 4\sigma(F_o)]$.

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

Mn(1)–O(4)#1	2.095(3)	O(5)#2–Mn(1)–O(1)	92.38(9)
Mn(1)–O(5)#2	2.108(3)	O(2)#3–Mn(1)–O(1)	89.25(9)
Mn(1)–O(2)#3	2.179(2)	O(2)–Mn(1)–O(1)	91.54(9)
Mn(1)–O(2)	2.194(2)	O(6)–Mn(1)–O(1)	179.70(16)
Mn(1)–O(6)	2.212(4)	O(3)#4–Mn(2)–O(3)	86.72(12)
Mn(1)–O(1)	2.2260(17)	O(3)#4–Mn(2)–O(3)#5	86.72(12)
Mn(2)–O(3)#4	2.160(3)	O(3)–Mn(2)–O(3)#5	86.72(12)
Mn(2)–O(3)	2.160(3)	O(3)#4–Mn(2)–O(1)#4	102.07(9)
Mn(2)–O(3)#5	2.160(3)	O(3)–Mn(2)–O(1)#4	94.17(9)
Mn(2)–O(1)#4	2.292(2)	O(3)#5–Mn(2)–O(1)#4	171.20(10)
Mn(2)–O(1)#5	2.292(2)	O(3)#4–Mn(2)–O(1)#5	94.17(9)
Mn(2)–O(1)	2.292(2)	O(3)–Mn(2)–O(1)#5	171.20(10)
Mn(2)–Mn(2)#6	3.1835(19)	O(1)#4–Mn(2)–O(1)#5	77.08(8)
		O(3)#4–Mn(2)–O(1)	171.20(10)
O(4)#1–Mn(1)–O(5)#2	3.1835(19)	O(3)–Mn(2)–O(1)	102.07(9)
O(4)#1–Mn(1)–O(2)#3	162.26(11)	O(3)#5–Mn(2)–O(1)	94.17(9)
O(5)#2–Mn(1)–O(2)#3	89.99(10)	O(1)#4–Mn(2)–O(1)	77.08(8)
O(4)#1–Mn(1)–O(2)	85.48(11)	O(1)#5–Mn(2)–O(1)	77.08(8)
O(5)#2–Mn(1)–O(2)	166.15(10)	O(3)#4–Mn(2)–Mn(2)#6	127.55(8)
O(2)#3–Mn(1)–O(2)	76.78(9)	O(3)–Mn(2)–Mn(2)#6	127.55(8)
O(4)#1–Mn(1)–O(6)	88.66(15)	O(3)#5–Mn(2)–Mn(2)#6	127.56(8)
O(5)#2–Mn(1)–O(6)	87.92(15)	O(1)#4–Mn(2)–Mn(2)#6	46.01(5)
O(2)#3–Mn(1)–O(6)	90.78(14)	O(1)#5–Mn(2)–Mn(2)#6	46.01(5)
O(2)–Mn(1)–O(6)	88.17(15)	O(1)–Mn(2)–Mn(2)#6	46.01(5)
O(4)#1–Mn(1)–O(1)	91.23(10)		

Note: Symmetry code: #1 $x - y, x, -z$; #2 $-y + 1/3, -x + 2/3, z + 1/6$; #3 $y + 1/3, x - 1/3, -z + 1/6$; #4 $-y + 1, x - y, z$; #5 $-x + y + 1, -x + 1, z$; #6 $x - y + 1/3, -y + 2/3, -z + 1/6$; #7 $y, -x + y, -z$; #8 $-y + 2/3, -x + 1/3, z - 1/6$.

ω scan mode at 296(2) K. The total of 3830 including 2850 independent reflections ($R_{\text{int}} = 0.0596$) were collected from $2.23 < \theta < 28.7°$. Unit cell dimensions were obtained with least-squares refinements, and multiscan corrections were applied using SADABS [27]. The structure was solved by direct method and non-hydrogen atoms were obtained in successive difference Fourier syntheses [28]. Hydrogens of C were included in calculated positions and refined with fixed thermal parameters riding on their parent atoms. The final refinement was performed by full matrix least-squares with anisotropic thermal parameters for non-hydrogen atoms on F^2. Crystal parameters and structure refinement are summarized in table 1. Selected bond lengths and angles are summarized in table 2. In the structure, the coordinated solvents and counter cations are highly disordered; we cannot get a suitable model from the diffraction data even collecting data three times. The contribution of the disordered guests was subtracted from the reflection data by the Squeeze method, and the results are contained in a CIF file.

3. Results and discussion

3.1. *General characterizations*

In the IR spectrum of **1** (figure 1), absorptions from the skeletal vibrations of aromatic rings were at 1580 cm^{-1}. The characteristic bands of the carboxylate groups were at 1650 cm^{-1} for the asymmetric stretch and 1380 cm^{-1} for the symmetric stretch [6]. To investigate the thermal stability of **1**, TGA on its polycrystalline sample in N_2 was performed from 25 to 800 °C (figure 2). This compound started to decompose at 100 °C until 400 °C. The phase purity of as-synthesized samples of **1** has been characterized by powder X-ray diffraction (PXRD) (figure 3). The experimental PXRD pattern matched well with the result simulated from the single-crystal data, indicating pure phase of **1**. The difference in reflection intensities between the simulated and experimental patterns may be due to the variation in preferred orientation of the powder samples during the collection of the experimental PXRD data.

3.2. *Description of crystal structures*

Single-crystal X-ray diffraction analysis revealed that **1** crystallizes in the space group *R-3c* and possesses an extended 3-D framework structure. The prominent structural feature of **1**

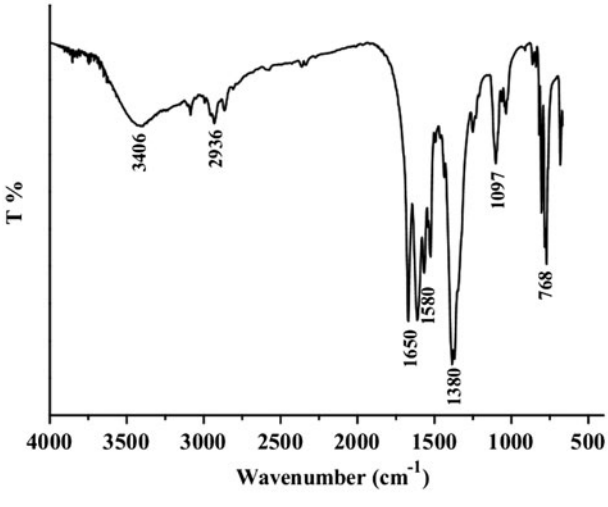

Figure 1. The IR spectrum of **1**.

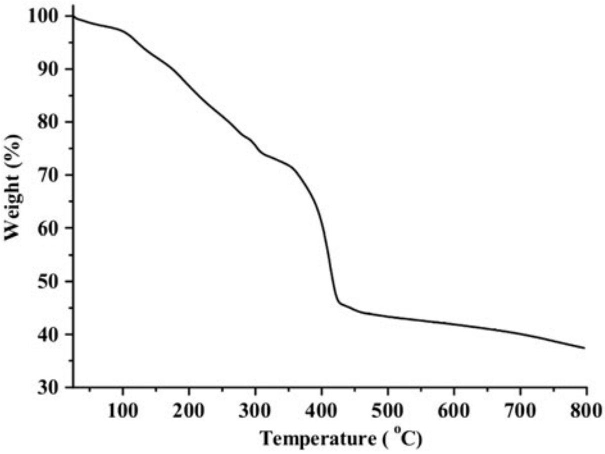

Figure 2. The TGA curve of **1**.

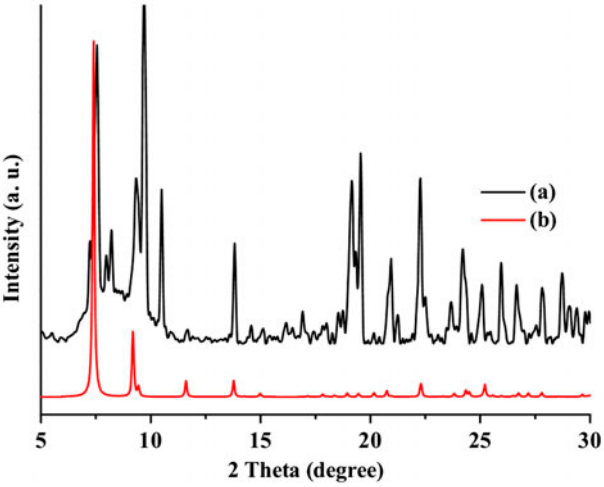

Figure 3. The PXRD patterns of **1**: (a, black) experimental; (b, red) simulated (see http://dx.doi.org/10.1080/00958972.2014.950255 for color version).

is the presence of octanuclear $Mn_8(\mu_4\text{-}O)_3(COO)_{12}$ SBU. As shown in figure 4(a), in the structure, there exist two crystallographically independent Mn ions. Mn1 adopts a distorted octahedral geometry, coordinated by six oxygens from four thb ligands; one from μ_4-O entity and one from coordinated solvent. Mn2 also adopts a distorted octahedral geometry, coordinated by six oxygens from three carboxylate groups of thb and three different μ_4-O entities. Six Mn1 and two Mn2 ions are bridged by three μ_4-O and 12 carboxylate groups to form an octanuclear $Mn_8(\mu_4\text{-}O)_3(COO)_{12}$ SBU [figure 4(b)]. The Mn–O distances in this SBU range from 2.0959(1) to 2.2922(1) Å. These SBUs are further connected by thb ligands to form an anionic 3-D framework with rhombic open channels [figure 4(d)]. Many Mn-based compounds with multinuclear Mn-based clusters acting as SBUs have been

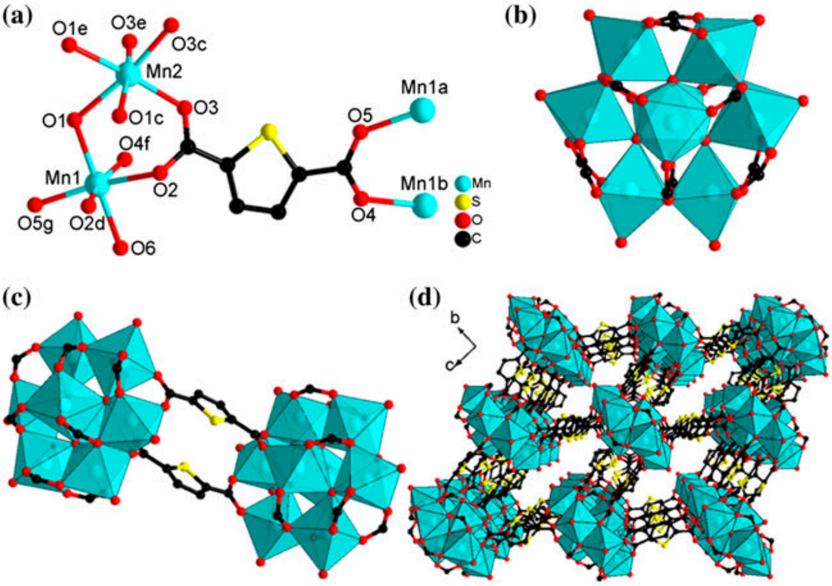

Figure 4. The structure of **1**. (a) the coordination environments of Mn ion and the thb ligand (symmetry codes: a: x, $1.5 - y$, $0.5 + z$; b: $2 - x$, $2 - y$ $1 - z$; c: x, $1.5 - y$, $-0.5 + z$; d: $1 - x$, $2 - y$, $1 - z$). (b) The 6-connected octanuclear $Mn_8(\mu_4\text{-}O)_3(COO)_{12}$ SBUs. (c) View of two octanuclear Mn_8 SBUs double linked by thb ligands. (d) View of the 3-D framework structure of **1** along the *a* axis.

reported, but MOFs containing highly symmetric SBUs with more than eight metal ions are rare [23–25]. The charge of the framework in **1** is balanced by $[(CH_3)_2NH_2]^+$ cations, derived from the decomposition of DMF [24, 29, 30]. However, these cations cannot be located from the X-ray diffraction due to their high disorder in the structure. In this

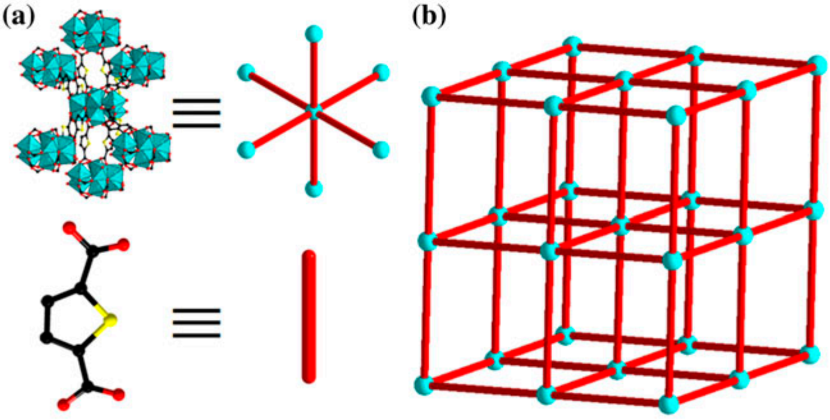

Figure 5. (a) The defined 6-connected nodes and lines. (b) Topological representation of two interpenetrating *pcu* nets in **1**.

structure, adjacent octanuclear SBUs are connected by two thb ligands as a double bridge to form the framework [figure 4(c)].

Topologically, each $Mn_8(\mu_4\text{-}O)_3(COO)_{12}$ SBU can be viewed as a 6-connected node, which is based on the fact that two adjacent SBUs are connected by two thb ligands as a linkage bridge [31, 32]. The thb ligand can be reduced as a linear linker [figure 5(a)]. Therefore, the framework of **1** can be simplified as a 6-connected *pcu* net as shown in figure 5(b).

3.3. *Magnetic properties of the complex*

Magnetic susceptibility measurements on microcrystalline sample of **1** were collected from 2 to 300 K under an applied field of 1000 Oe; corresponding curves of $\chi_M T$ and χ_M *versus* T are shown in figure 6. As the temperature decreases, the values of $\chi_M T$ remain nearly constant until 150 K and then drop to 4.86 cm^3 K M^{-1} at 2 K, revealing an overall antiferromagnetic coupling between the Mn(II) ions in **1** [33]. The susceptibility data above 10 K obey the Curie–Weiss law [$\chi_M = C/(T - \theta)$] with the parameters of $C = 37.30$ cm^3 K M^{-1} and $\theta = -21.19$ K (figure 7). The Curie value is a typical value of eight octahedral high-spin Mn(II) ions ($S = 5/2$, $g = 2.0$) [14]. The negative θ indicates dominant antiferromagnetic interaction between the Mn(II) ions. The absence of a peak in the χ_M *versus* T plot excludes any long-range antiferromagnetic order above 2 K, which is consistent with the structure of **1**. In the Mn_8 cluster of **1**, the Mn(II) ions are bridged by syn, syn, and μ_4-O, which conducted antiferromagnetic coupling between the Mn^{II} ions. The isothermal magnetization at 2 K (figure 8) increases with the field and reaches 20.43 $N\beta$ at 7 T, which is far from the saturation value of eight Mn(II) ions (40 $N\beta$). This supports the strong antiferromagnetic interactions between the Mn(II) ions in the cluster of **1**. The magnetic interactions in **1** are stronger than previously reported Mn-centered compound, $[Mn(L)(\mu 1,5\text{-dca})(CH_3OH)]_2$ (HL, (E)-3-hydroxy-N′-(pyridin-2-ylmethylene)-2naphthohydrazide) [34], and is comparable with a trinuclear Mn(IV) compound, which also shows strong antiferromagnetic coupling between metal centers [35]. No long-range antiferromagnetic order is found in **1**, being different from a MOF reported by Liu *et al.*, which shows long-range antiferromagnetic order as a peak in the χ_m *versus* T plot [36].

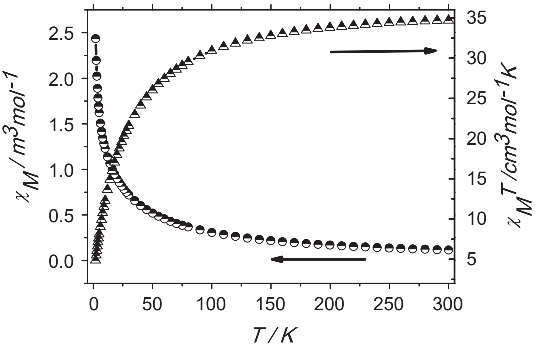

Figure 6. Plots of $\chi_M T$ *vs.* T and χ_M *vs.* T for **1** in the temperature range 2–300 K under applied field of 1000 Oe.

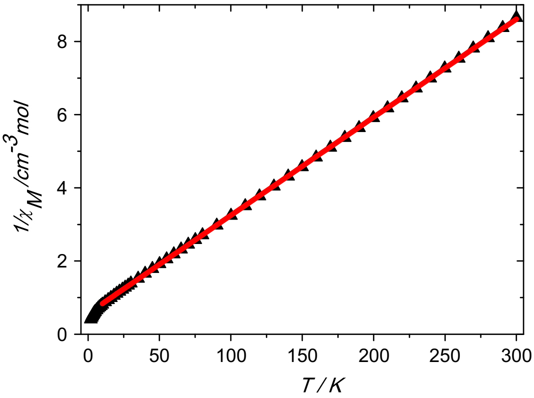

Figure 7. Plots of $1/\chi_M$ *vs.* T of **1**; the red solid line is best fitting by Curie–Weiss law.

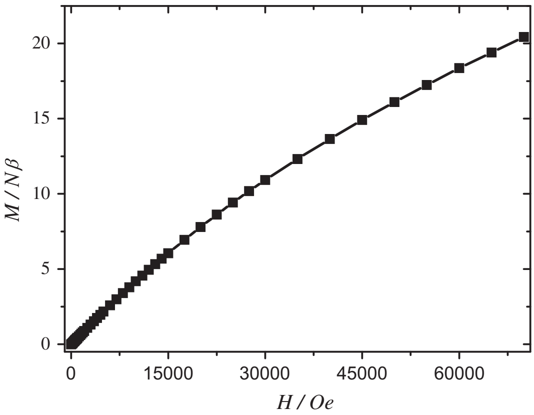

Figure 8. Field dependence of the magnetization for **1**.

4. Conclusion

A 3-D MOF based on $Mn_8(\mu_4\text{-}O)_3(COO)_{12}$ SBU has been solvothermally synthesized and structurally characterized. The high symmetry Mn_8 clusters are doubly linked by bridging dicarboxylate ligands to give the framework with a 6-connected *pcu* topology. Antiferromagnetic interactions were found between Mn(II) ions in the Mn_8 cluster of **1**.

Supplementary material

Crystallographic data for the structure reported in this article has been deposited on the Cambridge Crystallographic Data Center (CCDC No. 981156). The material can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html (or from the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK; Fax: +44 1223 336033; E-mail: deposit@ccdc.cam.ac.uk).

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