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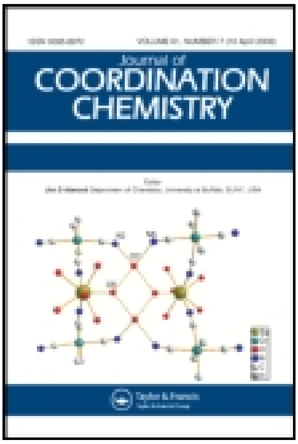

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Syntheses and structural characterization of two metal-organic frameworks from tripodal and dipodal ligands

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Syntheses and structural characterization of two metal-organic frameworks from tripodal and dipodal ligands

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Two isomorphic metal–organic frameworks, $\{[M_3(nbtb)_2(bpe)_3(H_2O)_2]\cdot 2H_2O\}_n$ (M = Co for (**1**) and Ni for (**2**)), have been synthesized by the combination of an extended linker 1,2-bi(4-pyridyl)ethene(bpe) and tripodal tecton 5-nitro-1,2,3-benzenetricarboxylic acid. Single-crystal X-ray diffraction studies indicate that **1** and **2** are (4, 5)-connected motifs containing $(4^2\cdot 6^7\cdot 8)$ $(4^2\cdot 6^7\cdot 8)$ $(6^4\cdot 8^2)$ $(4^2\cdot 6^4)$ topologies. The magnetic behavior of **1** exhibits weak antiferromagnetic interaction between metal centers.

Keywords: Metal–organic framework; Topology; Structure; Magnetism

1. Introduction

Integration of supramolecular and coordination chemistry gave rise to fascinating solid materials known as metal–organic frameworks (MOFs) [1–6]. A great number of metal–polycarboxylate compounds have been documented [7] with M attached through spacers to two/three carboxylic acid groups for binding with the metal, so-called dipodal and tripodal systems [8]. These solid materials form 1-D to 3-D motifs, depending on the mode of binding with metal. In contrast to classic metal–polycarboxylate MOFs, tripodal systems containing one or two organic substituents that can not coordinate to the metal could modulate the resultant structures. In addition, nonbonding groups may be positioned either away from or into any void with the MOFs. Recently, Zhou *et al*. [9] and Ma *et al*. [10] have studied the influence of the substituent on structures of MOFs. 5-Nitro-1,2,3-benzenetricarboxylic acid, as a derivative of 1,2,3-benzenetricarboxylic acid, has not been explored enough. In two isomorphic $\{[MNa_3(nbtb)(H_2O)_7]\cdot 4H_2O\}_n$ (M = Cu(II) and Ni(II)) complexes, Cu(II) is coordinated by six oxygens from five waters and one nbtb in a slightly distorted octahedral geometry and the Na(I) is surrounded by six waters in an irregular trigonal-prismatic geometry. The Cu(II) and Na(I) are linked by waters, forming 1-D infinite chains along the *a*-axis. In this study, we have prepared two isomorphic MOFs, $\{[M_3(nbtb)_2(bpe)_3(H_2O)_2]\cdot 2H_2O\}_n$ (M = Co for **1** and Ni for **2**), which represent

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Table 1. Crystallographic data for **1** and **2**.

Identification code	**1**	**2**
Empirical formula	$C_{54}H_{42}Co_3N_8O_{20}$	$C_{54}H_{42}N_8Ni_3O_{20}$
Formula weight	1299.75	1299.09
Temperature (K)	298(2)	298(2)
Crystal system	Triclinic	Triclinic
Space group	$P\bar{1}$	$P\bar{1}$
Unit cell dimensions (Å, °)		
a	13.014(2)	12.945(3)
b	13.659(2)	13.545(3)
c	15.836(3)	15.795(3)
α	94.959(2)	94.891(2)
β	90.090(2)	90.086(2)
γ	94.734(2)	94.808(2)
Volume (Å^3), Z	2794.5(8), 2	2749.6(9), 2
F(000)	1326	1332
Calculated density, ρ (g cm^{-3})	1.545	1.569
Goodness-of-fit	1.032	1.057
R_1, wR_2 [$I > 2\sigma(I)$]	0.0565, 0.1488	0.0529, 0.135
R_1, wR_2 (all data)	0.0851, 0.1708	0.0735, 0.1522
Residuals (e Å^{-3})	1.320, −0.704	1.420, −0.568

(4,5)-connected motifs containing $(4^2 \cdot 6^7 \cdot 8)$ $(4^2 \cdot 6^7 \cdot 8)$ $(6^4 \cdot 8^2)$ $(4^2 \cdot 6^4)$ topologies. The magnetism of **1** exhibits weak antiferromagnetic interaction between metal centers.

2. Experimental

2.1. *Materials and instruments*

All reagents used in the syntheses were of analytical grade. Elemental analyses for carbon, hydrogen, and nitrogen were performed on a Vario EL III elemental analyzer. Infrared (IR) spectra (4000 ~ 600 cm^{-1}) were recorded by using KBr pellets on an Avatar^{TM} 360 E. S. P. IR spectrometer. The crystal determinations were performed on a Bruker SMART APEX II CCD diffractometer equipped with graphite-monochromated Mo-Kα radiation ($\lambda = 0.71073$ Å). Variable-temperature magnetic susceptibility was measured using a MPMS-7 SQUID magnetometer. Diamagnetic corrections were made with Pascal's constants for all constituent atoms.

2.2. *X-ray crystallography*

Single-crystal X-ray diffraction analyses of the two compounds were carried out on a Bruker SMART APEX II CCD diffractometer equipped with graphite-monochromated Mo-Kα radiation ($\lambda = 0.71073$ Å) by using φ/ω scan technique at room temperature. The structures were solved by direct methods with SHELXS-97 [11]. 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 [12]. Table 1 shows crystallographic data of **1** and **2**. Selected bond distances and angles are listed in table 2. CCDC: 799282–799283.

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

1			
O(4)#1–Co(1)–O(2)	84.20(12)	O(10)–Co(1)–O(5)#1	81.83(12)
O(4)#1–Co(1)–O(10)	161.41(13)	O(4)#1–Co(1)–N(3)	103.16(13)
O(2)–Co(1)–O(10)	111.64(12)	O(2)–Co(1)–N(3)	88.27(13)
O(4)#1–Co(1)–O(5)#1	82.96(12)	O(10)–Co(1)–N(3)	87.51(13)
O(2)–Co(1)–O(5)#1	166.33(12)	O(9)–Co(2)–N(5)	92.18(13)
O(5)#1–Co(1)–N(3)	90.09(13)	O(6)#4–Co(3)–N(7)	95.01(13)
O(4)#1–Co(1)–N(4)#2	82.76(13)	O(11)#3–Co(2)–N(5)	98.38(13)
O(2)–Co(1)–N(4)#2	90.04(13)	O(13)#3–Co(2)–N(5)	90.32(13)
O(10)–Co(1)–N(4)#2	87.41(13)	O(1)–Co(2)–N(6)#2	83.88(13)
O(5)#1–Co(1)–N(4)#2	92.99(13)	O(9)–Co(2)–N(6)#2	86.20(13)
N(3)–Co(1)–N(4)#2	173.63(14)	O(11)#3–Co(2)–N(6)#2	85.86(13)
O(1)–Co(2)–O(9)	104.23(13)	O(13)#3–Co(2)–N(6)#2	91.88(13)
O(1)–Co(2)–O(11)#3	165.11(12)	N(5)–Co(2)–N(6)#2	175.35(14)
O(9)–Co(2)–O(11)#3	85.78(12)	O(18)–Co(3)–O(14)	87.33(13)
O(1)–Co(2)–O(13)#3	83.43(12)	O(18)–Co(3)–O(17)	171.69(13)
O(9)–Co(2)–O(13)#3	171.83(12)	O(14)–Co(3)–O(17)	93.44(13)
O(11)#3–Co(2)–O(13)#3	86.16(12)	O(18)–Co(3)–O(6)#4	92.03(13)
O(1)–Co(2)–N(5)	92.30(13)	O(14)–Co(3)–O(6)#4	169.47(13)
O(17)–Co(3)–O(6)#4	88.72(12)	O(18)–Co(3)–N(8)#2	94.52(14)
O(18)–Co(3)–N(7)	85.74(14)	O(14)–Co(3)–N(8)#2	85.13(13)
O(14)–Co(3)–N(7)	95.42(14)	O(17)–Co(3)–N(8)#2	93.79(14)
O(17)–Co(3)–N(7)	85.95(14)	O(6)#4–Co(3)–N(8)#2	84.45(13)
2			
Ni(1)–O(7)	2.073(3)	Ni(2)–O(18)#3	2.066(3)
Ni(1)–O(5)	2.085(3)	Ni(2)–N(1)#2	2.088(3)
Ni(1)–N(3)	2.088(3)	Ni(2)–N(2)	2.105(3)
Ni(1)–O(13)	2.106(3)	Ni(3)–O(10)#1	2.027(3)
Ni(1)–O(9)#1	2.112(3)	Ni(3)–O(15)#4	2.069(3)
Ni(1)–N(4)#2	2.122(3)	Ni(3)–O(14)	2.075(3)
Ni(2)–O(1)	2.024(3)	Ni(3)–N(6)	2.083(3)
Ni(2)–O(2)	2.059(3)	Ni(3)–O(17)#4	2.099(3)
Ni(2)–O(6)	2.064(3)	Ni(3)–N(5)#2	2.113(3)
O(7)–Ni(1)–O(5)	84.84(11)	O(1)–Ni(2)–O(2)	171.77(13)
O(7)–Ni(1)–N(3)	102.27(12)	O(1)–Ni(2)–O(6)	92.65(12)
O(5)–Ni(1)–N(3)	90.72(12)	O(2)–Ni(2)–O(6)	88.32(12)
O(7)–Ni(1)–O(13)	164.05(12)	O(1)–Ni(2)–O(18)#3	87.18(12)
O(5)–Ni(1)–O(13)	82.46(11)	O(2)–Ni(2)–O(18)#3	93.32(12)
N(3)–Ni(1)–O(13)	87.54(12)	O(6)–Ni(2)–O(18)#3	169.71(12)
O(7)–Ni(1)–O(9)#1	83.34(11)	O(1)–Ni(2)–N(1)#2	85.87(13)
O(5)–Ni(1)–O(9)#1	167.54(11)	O(2)–Ni(2)–N(1)#2	85.91(13)
N(3)–Ni(1)–O(9)#1	87.94(12)	O(6)–Ni(2)–N(1)#2	95.07(12)
O(13)–Ni(1)–O(9)#1	109.84(11)	O(18)#3–Ni(2)–N(1)#2	95.19(13)
O(7)–Ni(1)–N(4)#2	83.09(12)	O(1)–Ni(2)–N(2)	94.37(13)
O(5)–Ni(1)–N(4)#2	92.29(12)	O(2)–Ni(2)–N(2)	93.86(13)
N(3)–Ni(1)–N(4)#2	174.08(13)	O(6)–Ni(2)–N(2)	84.49(12)
O(13)–Ni(1)–N(4)#2	87.80(12)	O(18)#3–Ni(2)–N(2)	85.26(12)
O(9)#1–Ni(1)–N(4)#2	90.20(12)	N(1)#2–Ni(2)–N(2)	179.51(13)
N(6)–Ni(3)–O(17)#4	90.71(12)	O(10)#1–Ni(3)–O(15)#4	166.20(11)
O(10)#1–Ni(3)–N(5)#2	84.48(12)	O(10)#1–Ni(3)–O(14)	103.32(12)
O(15)#4–Ni(3)–N(5)#2	85.25(12)	O(15)#4–Ni(3)–O(14)	85.21(12)
O(14)–Ni(3)–N(5)#2	86.42(12)	O(10)#1–Ni(3)–N(6)	92.58(12)
N(6)–Ni(3)–N(5)#2	176.20(13)	O(15)#4–Ni(3)–N(6)	98.02(12)
O(17)#4–Ni(3)–N(5)#2	91.34(12)	O(14)–Ni(3)–N(6)	91.93(12)
O(14)–Ni(3)–O(17)#4	172.74(12)	O(10)#1–Ni(3)–O(17)#4	83.32(11)

Symmetry transformations used to generate equivalent atoms in **1**: #1 $-x+1$, $-y$, $-z+1$; #2 x, $y-1$, z; #3 $-x+1$, $-y$, $-z+2$; #4 $x-1$, y, $z+1$.

Symmetry transformations used to generate equivalent atoms in **2**: #1 $-x+1$, $-$y, $-z+1$; #2 x, $y-1$, z; #3 $-x$, $-y$, $-z+2$; #4 $-x+1$, $-y$, $-z+2$; #5 x, $y+1$, z.

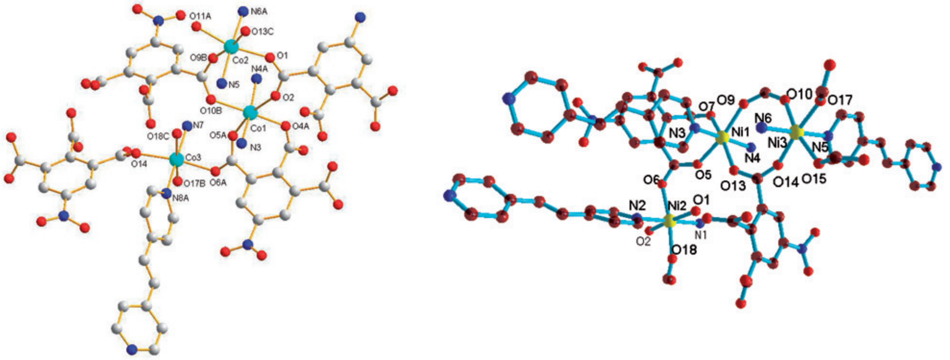

Figure 1. The coordinated geometries of Co(II) and Ni(II) in **1** and **2** (left for Co and right for Ni).

2.3. *Preparation of MOFs 1 and 2*

2.3.1. $\{[Co_3(nbtb)_2(bpe)_3(H_2O)_2]\cdot 2H_2O\}_n$ (1). A mixture of $Co(OAc)_2\cdot 4H_2O$ (125 mg, 0.5 mmol), H_3nbtb (26 mg, 0.1 mmol), and bpe (18.5 mg, 0.1 mmol) was stirred into 10 mL DMF-H_2O solution. Then the pH was adjusted to 6 with 1 mol L^{-1} NaOH, the reaction mixture heated on a water bath for 4 days at 150°C, and then filtered. Pink crystals separated after the solution cooled to room temperature. Anal. Calcd for $C_{54}H_{42}Co_3N_8O_{20}$ (%): C, 49.90; H, 3.26; N, 8.62. Found (%): C, 49.95; H, 3.11; N, 8.36. IR (KBr, cm^{-1}). 3455vs, 1588s, 1438s, 1076m, 917m.

2.3.2. $\{[Ni_3(nbtb)_2(bpe)_3(H_2O)_2]\cdot 2H_2O\}_n$ (2). **2** was synthesized in a procedure analogos to that of **1** except that $Ni(OAc)_2\cdot 4H_2O$ was used instead of $Co(OAc)_2\cdot 4H_2O$. Anal. Calcd for $C_{54}H_{42}Ni_3N_8O_{20}$ (%): C, 49.93; H, 3.23; N, 8.63. Found (%): C, 49.55; H, 3.41; N, 8.55. IR (KBr, cm^{-1}). 3502vs, 1601s, 1488s, 1176m, 902m.

3. Results and discussion

Single-crystal X-ray structural analysis shows that **1** and **2** are isostructural, and thus, only the crystal structure of **1** will be described herein. The structure of **1** presents a 3-D-pillared coordination polymer with a unique mixed-connected topology. The asymmetric unit consists of three Co(II)/Ni(II) ions, a pair of nbtb, three bpe, and four-coordinated/lattice waters (figure 1). The three Co(II) ions are six coordinate with distorted octahedral coordination geometries. The six donors coordinated to Co1/Co2 are two pyridyl nitrogens of two bpe linkers and four carboxylate oxygens from three nbtb. Co3 is coordinated by two waters, two carboxylato-O from nbtb, and two nitrogens from bpe. This coordination of nbtb is very similar to that of $\{[Mn_3(nbtb)_2(bipy)_3(H_2O)_2]\cdot 2H_2O\}_n$ [10]. Co1 and Co2 are connected by a pair of *syn–syn* carboxylate groups of two different nbtb ligands to provide a binuclear unit, and those binuclear units are further extended by nbtb linkages into 1-D metal chains,

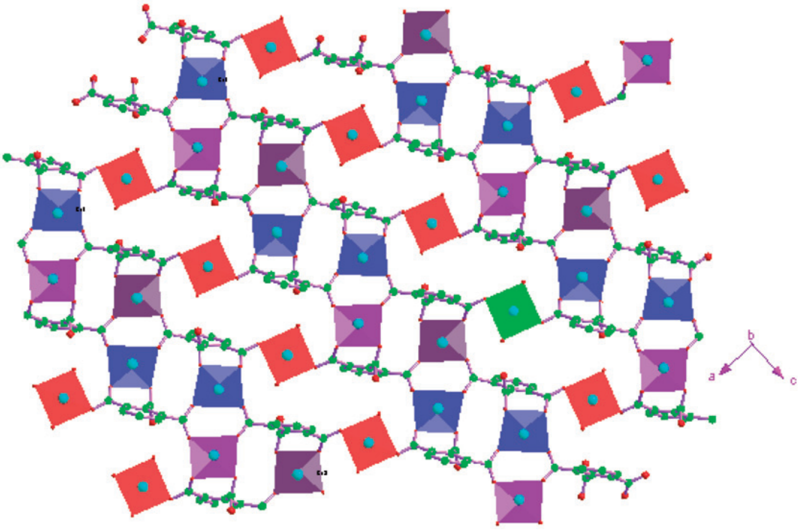

Figure 2. View of 2-D layer directed by Co(II) and nbtb (pink octagon is Co1; blue octagon is Co2; red octagon is Co3).

which are then connected by Co3 to give a 2-D grid layer (figure 2). The [Co(nbtb)] layers are constructed as coplanar aggregations of CoOCO four-membered rings and 32-membered $Co_4O_4C_4O_4$ elliptical circuits. The $Co \cdots Co$ distances through the bisbidentate-bridging segments of carboxylates are 4.45–8.51 Å. Individual [Co(nbtb)] layers are then pillared into a 3-D network by means of virtually flat bpe ligands oriented parallel to the *b* crystal axis (figure 3). To better understand the complicated motif of **1**, the topological approach is applied to simplify such a 3-D coordination framework. If, for reasons of classifying the net, we define chelating ring as a single point of connection to Co, then both Co1 and Co2 are depicted as five-connected nodes (green and pink spheres in figure 4), and Co3 and ntbt serve as four-connected nodes (blue and yellow spheres in figure 4). In this way, **1** can be reduced to a tetranodal (4, 5)-connected motif with the $(4^2 \cdot 6^7 \cdot 8)(4^2 \cdot 6^7 \cdot 8)(6^4 \cdot 8^2)(4^2 \cdot 6^4)$. The structure of $\{[Cd_3(nbtb)_2(bpe)_3(H_2O)_2] \cdot 2H_2O\}_n$ features a four-connected self-interpenetrating 3-D coordination net with $(4 \cdot 6^2 \cdot 7^2 \cdot 8)_2(4^2 \cdot 6^2 \cdot 7 \cdot 8)$ topology [13]. A comparison of $\{[Cd_3(nbtb)_2(bpe)_3(H_2O)_2] \cdot 2H_2O\}_n$ and **1** and **2** clearly indicates that the metal ions have a significant influence on the assembly of resultant nets. Waters of crystallization, which hydrogen bond to chelating nbtb, partially fill the void spaces.

Asymmetric ligands (such as 1,2,3-benzenetricarboxylic acid and 1,3,5-benzenetricarboxylic acid) with the multiplicity of the dihedral angle between the plane of the carboxylate and the phenyl ring plane have been used to bind metal centers in different directions to form many complexes with channels and cavities [14]. $[Zn_2(OH)(BTC)(bix)] \cdot H_2O$ and Zn(BIPA)(bix) (H_3BTC = benzene-1,2,3-tricarboxylic acid, H_2BIPA = 5-bromoisophthalic acid, and bix = 1,4-bis(imidazol-1-ylmethyl)benzene) exhibit 2-D (3,4)-connected and 2-D → 3-D parallel polycatenation nets, respectively. In a new Cu–BTC system, a new 3-D compound $\{[Cu_5(BTC)(4,4'\text{-bipy})_2(H_2O)_6][Cu(4,4'\text{-bipy})(H_2O)_4]\}_n \cdot 8nH_2O$ was obtained by diffusion method,

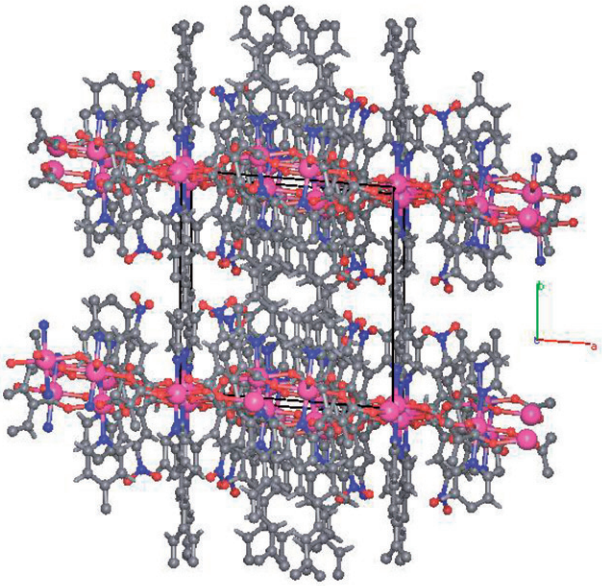

Figure 3. Perspective view of 3-D-pillared network in **1**.

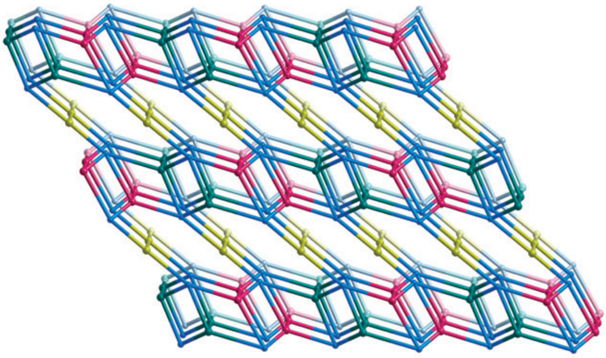

Figure 4. Schematic view of (4,5)-connected topology in **1** (green and pink spheres for Co1 and Co2 with 5-connected nodes; blue and yellow spheres for Co3 and nbtb ligands with 4-connected nodes).

which contains tetranuclear paddle-wheel clusters and presents strong fluorescence character. This phenomenon was also observed in other M-polycarboxylate systems with bridging N-donors. Thus, the ancillary ligand and metal ions have significant effects on the formation and structure of the coordination polymers.

3.1. *Magnetic properties of 1*

Magnetic susceptibility, χ_M, of **1** was measured in the 2–300 K temperature range. The magnetic data of **1** are displayed in figure 5, plotted as the thermal variation of

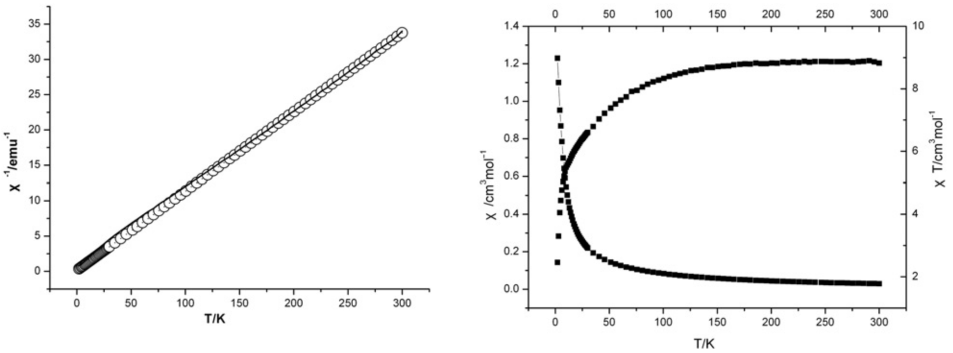

Figure 5. Plots χ_M^{-1} of *vs*. T (left); and χ_M and $\chi_M T$ of *vs*. T for **1**; solid lines represent fits to the data.

$\chi_M T$ and χ_M. The experimental $\chi_M T$ value at 300 K is 8.91 $cm^3\,mol^{-1}$ K, then decrease monotonically to 0.15 at 2 K. The inverse susceptibility $1/\chi_M$ plot as a function of temperature (T) is linear, closely following the Curie–Weiss law with $C = 8.92\,emu\,mol^{-1}\,K$, corresponding to about one $S = 3/2$ spin per formula unit [15]. The Weiss temperature, $\theta = -6.35\,K$, indicates that predominantly antiferromagnetic interactions exist between Co(II) centers. According to the structure of **1**, the main magnetic interactions between metal centers might happen in 1-D metal chains, while the superexchange interactions between Co ions through the nbtb bridge can be ignored owing to the length of bpe [16, 17]. As shown in figure 5, the calculated curve matches the experimental data quite well, the small deviations being most likely due to the fact that the Line theory was derived for six-coordinate Co(II) with O_h symmetry. The nature of magnetic coupling in **1** is the same as that observed in [Co(III)(μ-L_a)(μ-L_b)((μ-OAC))Co(II) · 2DMF (HL_b = salicylaldimine and HL_a = *N*-(2-hydroxybenzyl)salicylaldimine) [18a]. When the number of magnetic orbitals on each spin carrier is increased (from two in Ni(II) to three in Co(II)) the possibility of net overlap between the magnetic orbitals increases and antiferromagnetic terms associated to them counterbalance the countercomplementary effects and the magnetic interactions become antiferromagnetic in **1**, similar to {[Ni(μ-H_2O)(CH_3OH)(DFM)$(L)_2$] · H_2O · CH_3OH} (H_2L = *N,N*′-bis(5-ethyl-1,3,4-thiadiazol-2-yl)-2,6-pyridinedicarboxamide) and other related Ni(II) compounds [18b–18e].

4. Conclusion

Two isostructural coordination polymers based on rigid nbtb and bpe co-ligands have been prepared and characterized, with (4, 5)-connected motifs containing $(4^2 \cdot 6^7 \cdot 8)$ $(4^2 \cdot 6^7 \cdot 8)$ $(6^4 \cdot 8^2)$ $(4^2 \cdot 6^4)$ topologies. The magnetism of **1** exhibits weak antiferromagnetic interactions between metal centers. More metal complexes containing N-donor ligands and asymmetric aromatic polycarboxylates with interesting structures as well as physical properties will be synthesized.

Acknowledgment

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