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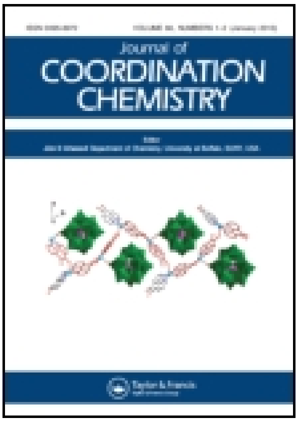

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Syntheses and structures of 3d-4d heterometallic coordination polymers based on 4-amino-1,2,4-triazole

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Syntheses and structures of 3d–4d heterometallic coordination polymers based on 4-amino-1,2,4-triazole

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Three heterometallic coordination polymers based on 4-amino-1,2,4-triazole (atrz), $Cd_{2.98}Fe_{2.02}(atrz)_6(SCN)_{10}\cdot 2H_2O$ (**1**), $Cd_{2.86}Co_{2.14}(atrz)_6(SCN)_{10}\cdot 2H_2O$ (**2**), and $Cd_{2.02}Ni_{2.98}(atrz)_6(SCN)_{10}\cdot 2H_2O$ (**3**), were synthesized. Crystal structures of the polymers were determined by single-crystal X-ray diffraction, X-ray powder diffraction, and infrared analysis. The complexes exhibit a 3-D structure and are isostructural, but entail different durations for transformation from a 1-D polymer to a 3-D structure. The results of vibrating sample magnetometry for **1** indicate that the complex exhibits weak paramagnetism.

Keywords: Heterometallic; Coordination polymers; Magnetism

1. Introduction

Metal-organic coordination polymers (CPs) receive attention for intriguing architectures and physical and/or chemical properties, including ferroelectricity, luminescence, magnetism, nonlinear optics, and gas storage [1–5]. Usually, the structure of CPs is influenced by coordination geometries of metal [6, 7], and structural characteristics of the ligands [8, 9], and counter anions [10, 11]. Polynuclear transition metal complexes with derivatives of 1,2,4-triazole derivatives have elicited growing interest because of magnetic properties [12, 13]. A series of hybrid 3d–3d [14, 15] and 3d–4f [16, 17] metal complexes that exhibit interesting structures and significant properties have been synthesized.

In this paper, on the basis of previous studies on heterometallic complexes of Zn(II) and Cd(II) [18], we use 4-amino-1,2,4-triazole (atrz) and thiocyanate (SCN^-) as ligands and Fe(II)–Cd(II)/Co(II)–Cd(II) as metals to obtain two heterometallic complexes. Structural characterizations indicate that the complexes are 3-D structures, similar to hybrid metal complexes reported previously. If the coordinated metal is Cd, a 1-D chain polymer $[Cd(atrz)_2(SCN)_2]_n$ is synthesized, as reported in previous research [19].

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2. Experimental

2.1. *Materials and instruments*

4-Amino-1,2,4-triazole (atrz) was prepared by the literature method [20]. Other reagents were obtained from commercial sources and used without purification. The crystal determinations were performed on a RAXIS SPIDER diffractometer equipped with graphite-monochromated Mo $K\alpha$ radiation ($\lambda = 0.71073$ Å). Powder crystal X-ray diffraction (XRD) measurements were carried out on a Bruker D8 AVANCE. Elemental analyses were carried out on a Thermo Flash EA-1112. Infrared (IR) spectra (KBr) were recorded on a Bruker EQUINOX 55 spectrometer from 400–4000 cm^{-1}. Atomic absorption was performed by a Hitachi Z-2000. Magnetic susceptibility was carried out on a VSM Lakeshore 7404.

2.2. *X-ray crystallography*

X-ray single-crystal diffraction was determined at 293 ± 2 K. The structure was solved by direct methods and refined on F^2 by full-matrix least-squares with SHELXTL-97 [21]. All nonhydrogen atoms were refined anisotropically. Hydrogens of atrz were localized in their calculated positions and refined using a riding model. Hydrogens of waters were not treated. Table 1 shows crystallographic data of **1** and **2**. Selected bond distances and angles are listed in table 2, CCDC: 901670–901671.

2.3. *Preparation of complexes*

2.3.1. $Cd_{2.98}Fe_{2.02}(atrz)_6(SCN)_{10}\cdot 2H_2O$ (1). A mixture of $Cd(NO_3)_2\cdot 4H_2O$ (0.46 g, 1.5 mmol), $FeSO_4\cdot 7H_2O$ (0.42 g, 1.5 mmol), and NH_4SCN (0.61 g, 8.0 mmol) was stirred in a 20 mL aqueous solution for 30 min at 80 °C. Then, atrz (0.25 g, 3 mmol) dissolved in

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

Compound	**1**	**2**
Empirical formula	$C_{22}H_{28}Cd_{2.98}Fe_{2.02}N_{34}O_2S_{10}$	$C_{22}H_{28}Cd_{2.86}Co_{2.14}N_{34}O_2S_{10}$
Formula weight	1569.15	1554.84
Temperature (K)	293(2)	29 (2)
Crystal system	Monoclinic	Monoclinic
Space group	$P\ 2(1)/n$	$P\ 2(1)/n$
a	11.233(2)	11.101(2)
b	15.143(3)	15.172(3)
c	15.789(3)	15.672(3)
β	96.29(3)	95.80(3)
Volume (Å^3), Z	2669.4(9), 2	2626.1(9), 2
$F(000)$	1539	1538
Calculated density (g/cm^3)	1.952	1.985
Goodness-of-fit on F^2	1.128	1.180
R_1, wR_2 [I>2sigma(I)]	0.0498, 0.1355	0.0611, 0.1664
R_1, wR_2 (all data)	0.0642, 0.1610	0.0857, 0.2352
Largest diff. peak hole (e Å^{-3})	1.459, −1.004	1.501, −1.964

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

1			
Cd(1)–N(16)	2.285(6)	N(10)#1–M(2)–N(10)	180.0(3)
Cd(1)–S(6)	2.613(2)	N(10)#1–M(2)–N(6)#1	89.2(2)
Cd(1)–S(4)	2.684(2)	N(10)–M(2)–N(6)#1	90.8(2)
Cd(1)–S(8)	2.730(2)	N(10)–M(2)–N(6)#1	90.8(2)
Cd(1)–S(5)	2.819(2)	N(10)–M(2)–N(6)	89.2(2)
Cd(1)–S(7)	2.826(2)	N(6)#1–M(2)–N(6)	180.0(3)
M(2)–N(10)#1	2.247(6)	N(10)#1–M(2)–N(13)#1	90.4(2)
M(2)–N(10)	2.247(6)	N(10)–M(2)–N(13)#1	89.6(2)
M(2)–N(6)#1	2.274(6)	N(6)#1–M(2)–N(13)#1	90.8(2)
M(2)–N(6)	2.274(6)	N(6)–M(2)–N(13)#1	89.2(2)
M(2)–N(13)#1	2.289(6)	N(10)#1–M(2)–N(13)	89.6(2)
M(2)–N(13)	2.289(6)	N(10)–M(2)–N(13)	90.4(2)
M(3)–N(1)	2.142(7)	N(6)#1–M(2)–N(13)	89.2(2)
M(3)–N(2)	2.151(7)	N(6)–M(2)–N(13)	90.8(2)
M(3)–N(5)	2.210(6)	N(13)#1–M(2)–N(13)	180.0(7)
M(3)–N(4)	2.218(6)	N(1)–M(3)–N(5)	97.2(2)
M(3)–N(3)	2.219(7)	N(2)–M(3)–N(5)	92.6(3)
M(3)–N(9)	2.222(5)	N(1)–M(3)–N(4)	89.1(2)
N(16)–Cd(1)–S(6)	169.0 (2)	N(2)–M(3)–N(4)	176.0(2)
N(16)–Cd(1)–S(4)	97.5(2)	N(1)–M(3)–N(2)	86.9(3)
S(6)–Cd(1)–S(4)	93.40(7)	N(5)–M(3)–N(4)	88.1(2)
N(16)–Cd(1)–S(8)	87.8 (2)	N(1)–M(3)–N(3)	94.0(3)
S(6)–Cd(1)–S(8)	91.83(8)	N(2)–M(3)–N(3)	92.3(3)
S(4)–Cd(1)–S(8)	99.35(7)	N(5)–M(3)–N(3)	168.0(2)
N(16)–Cd(1)–S(5)	81.4(2)	N(4)–M(3)–N(3)	87.7(2)
S(6)–Cd(1)–S(5)	97.70(8)	N(1)–M(3)–N(9)	177.2(2)
S(4)–Cd(1)–S(5)	87.81(6)	N(2)–M(3)–N(9)	92.0(2)
S(8)–Cd(1)–S(5)	167.75(7)	N(3)–M(3)–N(9)	83.5(2)
N(16)–Cd(1)–S(7)	88.2(2)	C(9)–N(13)–M(2)	131.0(5)
S(6)–Cd(1)–S(7)	80.91(7)	N(4)#1–N(13)–M(2)	122.2(4)
S(4)–Cd(1)–S(7)	165.75(7)	C(1)–N(9)–M(3)	122.3(4)
S(8)–Cd(1)–S(7)	93.90(6)	C(11)#3–S(8)–Cd(1)	102.1(3)
C(12)#2–S(4)–Cd(1)	95.2(3)	C(6)–S(7)–Cd(1)	106.1(3)
C(5)–S(5)–Cd(1)	99.2(3)	N(5)–M(3)–N(9)	85.4(2)
S(5)–Cd(1)–S(7)	80.09(6)	N(4)–M(3)–N(9)	92.0(2)
C(13)–S(6)–Cd(1)	99.5(3)		
2			
Cd(1)–N(17)	2.288(8)	N(2)#1–M(2)–N(2)	180.0(3)
Cd(1)–S(6)	2.621(3)	N(2)#1–M(2)–N(12)	90.1(3)
Cd(1)–S(4)	2.681(2)	N(2)–M(2)–N(12)	89.9(3)
Cd(1)–S(8)	2.719(3)	N(2)#1–M(2)–N(12)#1	89.9(3)
Cd(1)–S(7)	2.803(3)	N(2)–M(2)–N(12)#1	90.1(3)
Cd(1)–S(5)	2.830(3)	N(12)–M(2)–N(12)#1	180.0(4)
M(2)–N(2)#1	2.147(7)	N(2)#1–M(2)–N(6)	91.7(2)
M(2)–N(2)	2.147(7)	N(2)–M(2)–N(6)	88.3(2)
M(2)–N(12)	2.162(7)	N(12)–M(2)–N(6)	90.8(2)
M(2)–N(6)	2.176(6)	N(12)#1–M(2)–N(6)	89.2(2)
M(2)–N(6)#1	2.176(6)	N(2)#1–M(2)–N(6)#1	88.3(2)
M(3)–N(16)	2.080(8)	N(2)–M(2)–N(6)#1	91.7(2)
M(3)–N(9)	2.115(7)	N(12)–M(2)–N(6)#1	89.2(2)
M(3)–N(5)	2.148(7)	N(12)#1–M(2)–N(6)#1	90.8(2)
M(3)–N(11)	2.152(6)	N(6)–M(2)–N(6)#1	180.0(4)
M(3)–N(1)	2.156(6)	N(16)–M(3)–N(9)	86.7(3)
M(2)–N(12)#1	2.162(7)	N(16)–M(3)–N(5)	97.5(3)
M(3)–N(10)	2.173(8)	N(9)–M(3)–N(5)	92.7(3)
N(17)–Cd(1)–S(6)	167.2(2)	N(16)–M(3)–N(11)	90.0(3)
N(17)–Cd(1)–S(4)	98.8(2)	N(9)–M(3)–N(11)	176.8(3)

(*Continued*)

Table 2. (*Continued*)

S(6)–Cd(1)–S(4)	93.89(8)	N(5)–M(3)–N(11)	87.5(3)
N(17)–Cd(1)–S(8)	87.9(2)	N(16)–M(3)–N(1)	177.2(3)
S(6)–Cd(1)–S(8)	91.7(1)	N(9)–M(3)–N(1)	92.1(3)
S(4)–Cd(1)–S(8)	98.47(8)	N(5)–M(3)–N(1)	85.1(3)
N(17)–Cd(1)–S(7)	81.5(2)	N(11)–M(3)–N(1)	91.1(3)
S(6)–Cd(1)–S(7)	97.6(1)	N(16)–M(3)–N(10)	92.6(3)
S(4)–Cd(1)–S(7)	87.42(8)	N(9)–M(3)–N(10)	91.7(3)
S(8)–Cd(1)–S(7)	168.61(8)	N(5)–M(3)–N(10)	169.2(3)
N(17)–Cd(1)–S(5)	87.4(2)	N(11)–M(3)–N(10)	88.6(3)
S(6)–Cd(1)–S(5)	79.90(8)	N(1)–M(3)–N(10)	84.9(3)
S(4)–Cd(1)–S(5)	166.10(8)	C(3)–N(1)–M(3)	122.7(6)
S(8)–Cd(1)–S(5)	94.17(8)	N(2)–N(1)–M(3)	128.9(5)
S(7)–Cd(1)–S(5)	81.15(7)	C(5)–N(12)–M(2)	129.6(6)
C(10)#2–S(4)–Cd(1)	94.4(3)	N(11)#1–N(12)–M(2)	124.7(5)
C(9)–S(5)–Cd(1)	107.0(3)	C(11)–N(9)–M(3)	164.0(7)
C(8)–S(6)–Cd(1)	100.8(4)	C(1)–N(5)–M(3)	126.3(6)
C(7)–S(7)–Cd(1)	98.4(3)	N(6)–N(5)–M(3)	125.6(4)
C(11)#3–S(8)–Cd(1)	101.5(3)	C(4)–N(2)–M(2)	130.7(5)
C(10)–N(17)–Cd(1)	161.0(7)	N(1)–N(2)–M(2)	122.6(5)
C(2)–N(6)–M(2)	127.1(6)	C(6)#1–N(11)–M(3)	123.7(6)
N(5)–N(6)–M(2)	126.4(5)	N(12)#1–N(11)–M(3)	128.1(5)
C(7)–N(16)–M(3)	176.5(8)	C(9)#5–N(10)–M(3)	145.9(7)

Symmetry transformations used to generate equivalent atoms in **1**: #1 $-x$, $-y$, $-z$; #2 $-x+1$,$-y+1$,$-z$; #3 $x+1/2$,$-y+1/2$, $z-1/2$; #4 $x+1$, y, z; #5 $x-1$, y, z; #6 $x-1/2$, $-y+1/2$, $z+1/2$; in **2**: #1 $-x$, $-y$, $-z$; #2 $-x+1$, $-y+1$, $-z$; #3 $x+1/2$, $-y+1/2$, $z-1/2$; #4 $x+1$, y, z; #5 $x-1$, y, z; #6 $x-1/2$, $-y+1/2$, $z+1/2$.

10 mL methanol was added to the mixture. After refluxing for 3 h, the red solution was filtered and the filtrate was allowed to stand for evaporation at room temperature. Colorless block single crystals suitable for X-ray analysis were obtained after 3d. Anal. Calcd for $C_{22}H_{28}Cd_{2.98}Fe_{2.02}N_{34}OS_{10}$ (%): C, 16.84; H, 1.80; N, 30.35; S, 19.68; O, 2.04. Found (%): C, 16.70; H, 1.72; N, 29.94; S, 19.32; O, 1.95. AAS. Calcd (%): Cd, 21.35; Fe, 7.19. Found (%): Cd, 21.47; Fe, 7.03. IR (KBr, cm^{-1}): 3311m, 3088m, 2099vs, 1619m, 1211m, 993s, 619vs.

2.3.2. $Cd_{2.86}Co_{2.14}(atrz)_6(SCN)_{10}\cdot 2H_2O$ (2). **2** was synthesized in a procedure analogous to that of **1** except that $Co(NO_3)_2\cdot 6H_2O$ was used instead of $FeSO_4\cdot 7H_2O$. Pink lozenge-shaped single crystals suitable for X-ray analysis were obtained after 2d. Anal. Calcd for $C_{22}H_{28}Cd_{2.86}Co_{2.14}N_{34}OS_{10}$ (%): C, 16.98; H, 0.90; N, 30.61; S, 20.58; O, 2.06. Found (%): C, 16.70; H, 1.80; N, 30.53; S, 20.47; O, 1.99. AAS. Calcd (%): Cd, 20.68; Co, 8.11. Found (%): Cd, 20.80; Co, 8.02. IR (KBr, cm^{-1}): 3312m, 3057m, 2092vs, 1613m, 1211m, 997s, 621vs.

2.3.3. $Cd_{2.02}Ni_{2.98}(atrz)_6(SCN)_{10}\cdot 2H_2O$ (3). **3** was prepared in a procedure analogous to that of **1** except that $Ni(NO_3)_2\cdot 6H_2O$ was used instead of $FeSO_4\cdot 7H_2O$. Mauve single crystals were obtained after 1.5d, but the crystals were too small to be characterized by single-crystal XRD. Powder crystal determination indicates that the structure of **3** is similar to that of the first two complexes. Obtaining suitable crystals necessitates allowing complexes to evaporate for several days. Anal. Calcd for $C_{22}H_{28}Cd_{2.02}Ni_{2.98}N_{34}OS_{10}$ (%): C, 17.53;

H, 1.87; N, 31.59; S, 21.29; O, 1.06. Found (%): C, 17.50; H, 1.89; N, 31.50; S, 21.28; O, 1.14. AAS. Calcd (%): Cd, 15.06; Ni, 11.60. Found (%): Cd, 15.02; Ni, 11.69. IR (KBr, cm^{-1}): 3308m, 3055m, 2090vs, 1610m, 1212m, 1002s, 619vs.

3. Results and discussion

Single-crystal X-ray analysis reveals that **1**, **2**, and **3** are isostructural, exhibiting a 3-D structure. Thus, only the crystal structure of **1** is described in this section. The unit cell consists of five metal ions, six atrz ligands, ten SCN^-, and no H_2O coordinates to metal ions (figure 1(a)). Cd(II) and Fe(II) are positionally disordered with a ratio of 2.98 : 2.02. M2, M3, and M3A are bridged by six atrz ligands and form a trinuclear metal unit (figure 1 (b)). M2 is coordinated to six nitrogens from six different atrz ligands. Both M3 and M3A are coordinated to six nitrogens, three from three SCN^- and three from three different atrz. Cd and Cd1 are connected to the trinuclear metal unit of SCN^- and coordinated to five additional SCN^-. The M2-N distances range from 2.247(6) to 2.289(6) Å (average band distance 2.27(2) Å). This shows that M2 contains two kinds of metals (Cd and Fe), and the structure after analysis indicates that a small amount of iron exists in M2. The M3-N distances range from 2.142(7) to 2.222(5) Å (average band distance is 2.19(4) Å). Similarly, M3 consists of Fe and Cd, but only a small amount of Cd. M3A is similar to M3 because of their symmetrical structures. Therefore, the trinuclear unit may be presented as 3d–4d–3d. The magnetic 3d metals are separated by nonmagnetic 4d metal ions. Thus, the three complexes exhibit weak paramagnetism, in accord with the results of vibrating sample magnetometry.

3.1. *Study of the reaction mechanism*

The structures of the three complexes were characterized by powder XRD and IR (Supplementary material), indicating that the three isostructural heterometallic CPs are synthesized and that each complex is homogeneous.

When only Cd(II) is present, a 1-D polymer chain forms. However, when Fe, Co, Ni, and Zn with smaller radii are added the structure of the complexes changes into a 3-D structure. The structural transformation is not a casual occurrence, and a controllable regular pattern showing metal ion influence on the structure of complexes is observed.

When a different 3d metal is mixed, the duration of the structural transformation differs. The durations of the crystallization of **1**, **2**, and **3** are 3, 2, and 1.5 d, respectively. When Zn(II) is added, the crystallization entails 3d, indicating that different coordination properties of 3d metal ions can influence the crystallization process. The result may be attributed to the different rates of ligand exchange between metal ions. Fe(II) and Zn(II), especially Zn(II), are very easy to hydrate. Thus, the order of the length of time for crystallization follows the order: Zn(II) > Fe(II) > Co(II) > Ni(II).

3.2. *Magnetic properties of 1*

Powder samples of **1** were studied to determine magnetic properties. The temperature dependence of the magnetic susceptibility of **1**, in the form of χ_M and $\chi_M T$, is shown in figure 2, where χ_M is the magnetic susceptibility per trinuclear (M3, M2, M3A)

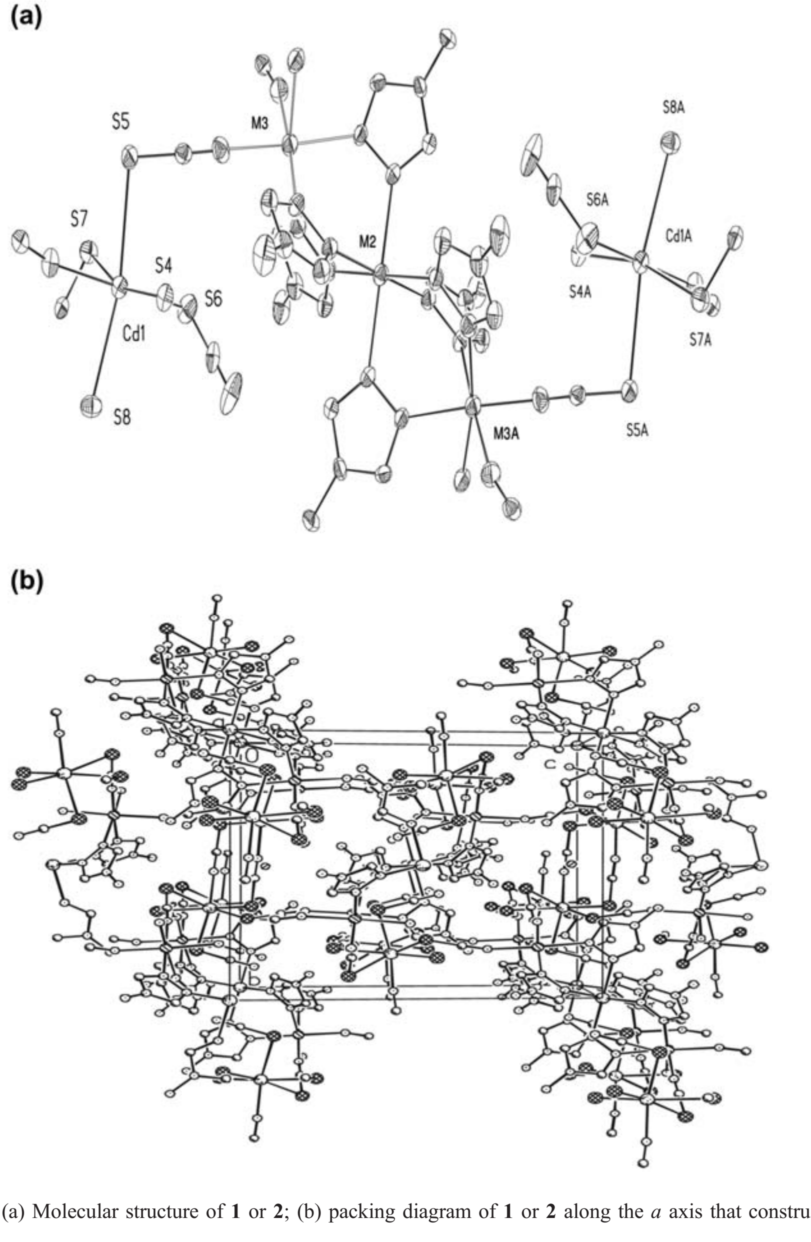

Figure 1. (a) Molecular structure of **1** or **2**; (b) packing diagram of **1** or **2** along the *a* axis that constructs a 3-D framework.

compound. The room temperature $\chi_M T$ is $4.5\,cm^3\,K\,mol^{-1}$, which is slightly larger than the expected value for two magnetically isolated Fe(II) (M3 and M3A) ions ($4.4\,cm^3\,K\,mol^{-1}$). $\chi_M T$ rapidly increases at 100 K to 290 K, but the χ_M value gradually decreases and reaches $3.15\,cm^3\,K\,mol^{-1}$ at 290 K. This magnetic behavior reveals weak paramagnetic interactions between the two Fe(II) centers and shows that the ground state is $S = 1$.

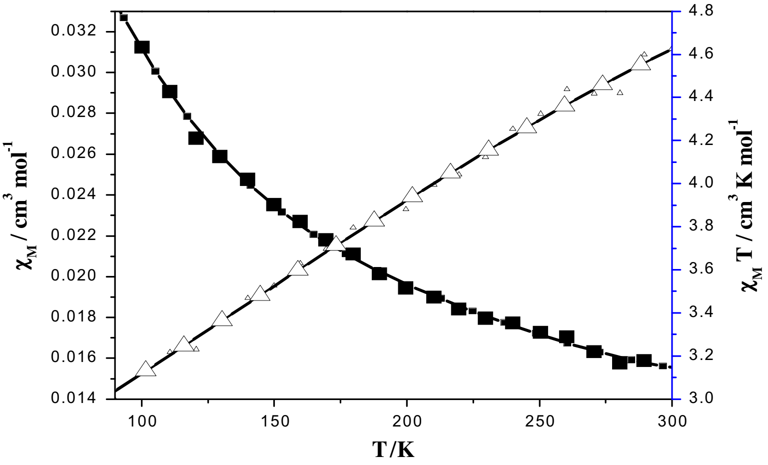

Figure 2. Plots of χ_M (■) and $\chi_M T$ (△) *vs*. temperature for **1**; the solid line represents the theoretical curve.

4. Conclusion

Two isostructural heterometallic CPs based on 4-amino-1,2,4-triazole and SCN$^-$ were synthesized and characterized. When Ni(II) is used instead of Fe(II)/Cd(II), a third isostructural heterometallic CP is obtained. All of the polymers are multinuclear and are based on 3-D frameworks.

Acknowledgments

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