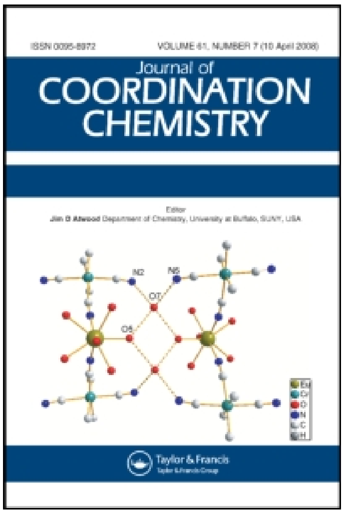

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Synthesis, structure and properties of a binuclear complex {[Mn(bpy)$_2$(CuL)(H$_2$O)$_2$](ClO$_4$)$_2$} and its CuL precursor (H$_2$L = 2,3-dioxo-5,6 : 14,15-dichlorobenzo-7,13-diphenyl-1,4,8,12-tetraazacyclo-pentadeca-7,12-diene)

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A binuclear complex {[Mn(bpy)$_2$(CuL)(H$_2$O)$_2$](ClO$_4$)$_2$} (**2**) and its CuL (**1**) precursor (H$_2$L = 2,3-dioxo-5,6 : 14,15-dichlorobenzo-7,13-diphenyl-1,4,8,12-tetraazacyclo-pentadeca-7, 12-diene; bpy = 2,2′-dipyridyl) have been synthesized and characterized structurally. Complex **1** consists of the neutral [CuL] fragments and methanol molecules. In complex **2**, [Mn(bpy)$_2$(CuL)]$^{2+}$ cation, two water molecules and two perchloric anions make up of the crystal. Mn(II) is coordinated by four nitrogen atoms from two 2,2′-dipyridyl and two oxygen atoms from the Cu(II) precursor. Magnetic characterization of **2** exhibits an antiferromagnetic interaction between Cu(II) and Mn(II) ions, with $J = -74.1\,\text{cm}^{-1}$.

Keywords: Macrocyclic oxamide complex; Complex ligand; Crystal structure; Magnetic properties

1. Introduction

Molecular magnetism and macrocyclic compound are two active fields of research encompassing chemistry, physics, biology and material science [1–4]. At the meeting point of these two fields, polynuclear macrocyclic oxamide complexes have received considerately attention in the design of new magnetic materials and for investigating the structure and the role of the polymetallic active sites in biology system [2–11]. In recent years, many oxamide-bridged polynuclear complexes containing macrocyclic ligand have been synthesized and studied magnetically because the oxamide group serves as a pathway through which election-spin interaction takes place [12–18]. In this article, we synthesized a macrocyclic complex [CuL] (**1**) (H$_2$L = 2,3-dioxo-5,6 : 14,15-dichlorobenzo-7,13-diphenyl-1,4,8,12-tetraazacyclo-pentadeca-7,12-diene; bpy = 2,2′-dipyridyl)

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and used it as a "complex ligand" to synthesize a hetero-binuclear complex $\{[Mn(bpy)_2(CuL)(H_2O)_2](ClO_4)_2\}$ (**2**). This novel "complex ligand" has never been used in previously literature.

2. Experimental

2.1. *Starting materials and synthesis of complex (1) (CuL)*

All the starting reagents are of A.R. grade and used as purchased. CuL was prepared by refluxing and stirring 2,2′-(oxalyldiimino)*bis*(chlorobenzaldehyde) (5.17 g, 0.01 mol), 1,3-propanediamine (1.6 mL, 0.02 mol), and $Cu(Ac)_2 \cdot H_2O$ (2.00 g, 0.01 mol) for 2 h in 50 mL of methanol with the presence of 4 drops of 2 M NaOH. After the mixture was cooled and filtered, the precipitate thus obtained was washed with water, methanol, and diethyl ether successively and dried under vacuum. The resulting deep green filtrate was kept at room temperature for several days, giving rise to green prism-like crystals suitable for X-ray analysis. Yield: 40%. Anal. Calcd: C, 57.64; H, 4.07; N, 14.15. Found: C, 57.92; H, 3.77; N, 14.44.

2.2. *Synthesis of complex (2)* $\{[Mn(bpy)_2(CuL)(H_2O)_2]\ (ClO_4)_2\}$

CuL (**1**) (0.062 g, 0.1 mmol) and Mn $(ClO_4)_2 \cdot 6H_2O$ (0.037 g, 0.1 mmol) were added into 50 mL methanol at the condition of stirring and slight heat of 80°C. After stirring for an hour, the methanol solution of 2,2′-dipyridyl was added dropwise. The mixture was stirred for 5 h and then cooled to room temperature and filtered. After two weeks or so, brownish-red crystals suitable for X-ray at room temperature were obtained from the filtrate. Yield: 32%. Anal. Calcd $C_{51}H_{42}Cl_4CuMnN_8O_{12}$: C, 50.24, H, 3.62, Cl, 11.7, Cu, 5.0, Mn, 4.2, N, 8.89, O, 15.93.

Caution! Perchlorate salts are potentially explosive and should only be handled in small quantities.

2.3. *Physical measurements*

Analyses of C, H and N were determined on a Perkin-Elmer 240 Elemental analyzer. IR spectrum was recorded as KBr discs on a Shimadzu IR-408 infrared spectrophotometer in the range of 4000–600 cm^{-1}. Magnetic susceptibilities were measured on a LDJ-9600 VSM between 80 and 300 K. Electronic spectra in DMF were recorded on a Shimadzu UV-2101 PC scanning spectrophotometer.

2.4. *X-ray crystallography*

The two complexes were mounted on a computer-controlled Bruker Smart 1000-CCD diffractometer equipped with graphite-monochromated Mo-Kα radiation ($\lambda = 0.71073$ Å).

Cell parameters were determined by the full matrix least-squares method on F^2 with anisotropic thermal parameters for all nonhydrogen atoms (SHELXL-97) [19].

Table 1. Crystal data and details of the structure determinations of complexes **1** and **2**.

	1	**2**
Empirical formula	$C_{32}H_{26}C_{l2}CuN_4O_3$	$C_{51}H_{42}Cl_4CuMnN_8O_{12}$
Formula weight	649.01	1219.21
Crystal system	Monoclinic	Orthorhombic
Space group	$P2(1)/c$	$Ibca$
a (Å)	11.016(4)	17.036(5)
b (Å)	16.349(6)	22.963(7)
c (Å)	16.756(7)	27.315(9)
α (°)	90	90
β (°)	97.906(7)	90
γ (°)	90	90
V (Å^3)	2989(2)	10,686(6)
Z	4	8
Absorption coefficient (mm^{-1})	0.950	0.904
$F(000)$	1332	4976
Crystal size (mm^3)	$0.24 \times 0.20 \times 0.18$	$0.22 \times 0.16 \times 0.12$
θ range for data collection (°)	2.24–26.40	2.11–25.00
Limiting indices	$-13 \leq h \leq 13$, $-18 \leq k \leq 20$, $-20 \leq l \leq 14$	$-20 \leq h \leq 14$, $-18 \leq k \leq 27$, $-32 \leq l \leq 28$
Reflections collected/unique	16,873/6099	24,063/4722
Max. and min. transmission	$[R_{(\mathrm{int})} = 0.0497]$ 1.000000 and 0.801322	$[R_{(\mathrm{int})} = 0.0637]$ 1.000000 and 0.794522
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
Data/restraints/parameters	6099/14/399	4722/73/386
Goodness-of-fit on F^2	1.047	1.043
Final R indices $[I > 2\sigma(I)]$	$R_1 = 0.0659$, $wR_2 = 0.1584$	$R_1 = 0.0572$, $wR_2 = 0.1397$
R indices (all data)	$R_1 = 0.1136$, $wR_2 = 0.1827$	$R_1 = 0.1135$, $wR_2 = 0.1797$
Largest diff. peak and hole (e Å^{-3})	1.145 and −0.236	0.683 and −0.405

Hydrogen atoms were added theoretically and refined with riding model position parameters and fixed isotropic thermal parameters. Further details of structure analysis are given in table 1. Position parameters and selected bond lengths and angles are listed in tables 2 and 3, respectively.

3. Results and discussion

3.1. *Crystal structure of complex 1*

The structure of complex **1** consists of the neutral fragment of [CuL] and two methanol molecules. A perspective view of complex **1** with labeling scheme is given in figure 1. The crystal data and refinement details are summarized in table 1. The dates of bond angles and bond lengths of complex **1** are showed in table 3. The macrocyclic ligand coordinates with the Cu(II) ion via two deprotonated oxamide nitrogens and two imine nitrogens. The deviation of the four nitrogen donors (N1, N2, N3, N4) from N4 are 0.3508, −0.3498, 0.3237, and −0.3248 Å, respectively, showing that the CuN4 chromophore assumes a tetrahedral coordination sphere. Figure 2 is the packing of complex **1**. There is a weak coordination bond between Cu(II) and oxygen of carbonyl of another molecule (Cu $\cdots$ O distance is 2.921 Å), which makes the crystal to be a 3D structure.

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters (Å$^2 \times 10^3$) (a) for complex **1**; (b) for complex **2**.

Atom	*x*	*y*	*z*	U(eq)
(a)				
Cu(1)	5631(1)	1441(1)	547(1)	40(1)
Cl(1)	−389(1)	1032(1)	1737(1)	88(1)
Cl(2)	10,855(2)	2306(1)	−1496(1)	83(1)
N(1)	4089(3)	938(2)	129(2)	38(1)
N(2)	6126(4)	1263(2)	−488(2)	39(1)
N(3)	6733(4)	2397(3)	770(2)	44(1)
N(4)	5312(3)	1378(2)	1651(2)	36(1)
O(1)	3369(4)	172(3)	−1011(2)	62(1)
O(2)	5271(4)	744(3)	−1719(2)	61(1)
O(3)	2754(13)	553(8)	7247(6)	126(4)
O(4)	3233(9)	1889(8)	6158(7)	108(4)
C(1)	4114(5)	626(3)	−620(3)	43(1)
C(2)	5268(5)	884(3)	−994(3)	43(1)
C(3)	7255(4)	1505(3)	−708(3)	39(1)
C(4)	7810(5)	1062(3)	−1288(3)	45(1)
C(5)	8898(5)	1294(4)	−1520(3)	54(2)
C(6)	9480(5)	1984(4)	−1184(3)	53(1)
C(7)	9008(5)	2414(3)	−601(3)	47(1)
C(8)	7904(4)	2180(3)	−337(3)	40(1)
C(9)	7505(5)	2667(3)	329(3)	44(1)
C(10)	8059(6)	3500(3)	460(4)	58(2)
C(11)	7707(7)	4103(4)	−103(5)	85(2)
C(12)	8190(12)	4891(6)	48(10)	148(6)
C(13)	8977(14)	5059(8)	729(11)	174(9)
C(14)	9335(11)	4474(7)	1265(7)	138(5)
C(15)	8883(8)	3684(5)	1133(5)	92(3)
C(16)	6371(5)	2877(4)	1443(3)	58(2)
C(17)	6752(6)	2425(4)	2237(3)	65(2)
C(18)	6449(5)	1519(4)	2206(3)	50(1)
C(19)	4280(4)	1233(3)	1910(3)	35(1)
C(20)	4227(4)	1225(3)	2802(3)	37(1)
C(21)	4138(5)	1944(4)	3215(3)	54(1)
C(22)	4113(6)	1929(4)	4040(4)	65(2)
C(23)	4163(6)	1206(5)	4443(4)	69(2)
C(24)	4253(6)	484(4)	4038(3)	63(2)
C(25)	4274(5)	489(3)	3211(3)	48(1)
C(26)	3136(4)	1073(3)	1361(3)	37(1)
C(27)	2048(4)	1102(3)	1722(3)	46(1)
C(28)	937(4)	990(3)	1271(4)	51(1)
C(29)	828(5)	833(3)	461(4)	51(1)
C(30)	1883(5)	782(3)	99(3)	47(1)
C(31)	3047(4)	917(3)	526(3)	37(1)
C(32)	3440(18)	1140(12)	6908(12)	135(7)
C(33)	1999(12)	2047(9)	5803(11)	102(5)
(b)				
Cu(1)	5000	2500	4576(1)	50(1)
Mn(1)	5000	2500	2583(1)	47(1)
Cl(1)	7379(1)	5116(1)	4333(1)	82(1)
Cl(2)	9140(2)	10,000	2500	74(1)
Cl(3)	7349(3)	2476(2)	4887(2)	85(2)
O(1)	5455(2)	2952(2)	3211(1)	60(1)
O(2)	9335(6)	10,583(3)	2319(4)	111(4)
O(3)	8583(9)	10,065(6)	2876(5)	133(6)
O(4)	9838(6)	9711(6)	2630(6)	195(8)
O(5)	8793(9)	9706(6)	2084(5)	144(7)
O(6)	7889(4)	2782(3)	4642(3)	157(3)

(*Continued*)

Table 2. Continued.

Atom	*x*	*y*	*z*	U(eq)
O(7)	7500	1888(4)	5000	184(5)
O(8)	6662(6)	2373(6)	4608(5)	131(4)
O(10)	6199(7)	1166(5)	4243(6)	134(4)
O(10′)	6326(7)	1781(6)	4205(6)	148(5)
N(1)	5415(2)	3001(2)	4061(1)	42(1)
N(2)	5123(3)	3109(2)	5073(1)	47(1)
N(3)	5958(3)	1833(2)	2479(2)	60(1)
N(4)	4117(3)	2110(2)	2076(2)	52(1)
C(1)	5253(3)	2780(2)	3628(2)	43(1)
C(2)	5873(3)	3501(2)	4129(2)	43(1)
C(3)	6258(3)	3781(2)	3738(2)	56(2)
C(4)	6715(4)	4264(3)	3798(2)	61(2)
C(5)	6818(3)	4490(2)	4256(2)	57(2)
C(6)	6450(3)	4241(2)	4652(2)	53(2)
C(7)	5958(3)	3754(2)	4603(2)	43(1)
C(8)	5536(3)	3575(2)	5047(2)	43(1)
C(9)	5579(3)	3970(2)	5485(2)	44(1)
C(10)	5087(4)	4444(3)	5523(2)	64(2)
C(11)	5088(4)	4778(3)	5946(3)	77(2)
C(12)	5579(5)	4648(3)	6321(2)	76(2)
C(13)	6080(4)	4191(3)	6280(2)	66(2)
C(14)	6084(3)	3848(2)	5867(2)	55(2)
C(15)	4640(4)	2984(3)	5512(2)	60(2)
C(16)	5000	2500	5820(3)	68(3)
C(17)	5991(5)	1324(3)	2722(3)	81(2)
C(18)	6663(7)	980(4)	2719(3)	99(3)
C(19)	7306(6)	1165(4)	2466(3)	102(3)
C(20)	7262(4)	1683(4)	2219(3)	86(2)
C(21)	6585(4)	2008(3)	2223(2)	60(2)
C(22)	4190(4)	1591(3)	1864(2)	66(2)
C(23)	3670(5)	1378(4)	1518(2)	86(2)
C(24)	3066(6)	1724(5)	1377(3)	99(3)
C(25)	2967(4)	2255(4)	1593(3)	88(2)
C(26)	3492(4)	2443(3)	1955(2)	59(2)

Table 3. Selected bond lengths (Å) and bond angles (°) for complexes **1** and **2**.

1			
Cu(1)–N(2)	1.910(4)	Cu(1)–N(4)	1.933(4)
Cu(1)–N(1)	1.930(4)	Cu(1)–N(3)	1.983(4)
N(2)–Cu(1)–N(1)	87.27(17)	N(2)–Cu(1)–N(3)	92.69(16)
N(2)–Cu(1)–N(4)	166.69(17)	N(1)–Cu(1)–N(3)	152.79(18)
N(1)–Cu(1)–N(4)	93.35(16)	N(4)–Cu(1)–N(3)	92.79(16)
2			
Cu(1)–N(1)	1.949(4)	O(1)–Mn(1)–N(4)#1	93.32(15)
Cu(1)–N(2)	1.961(4)	O(1)–Mn(1)–N(4)	158.76(17)
Mn(1)–O(1)	2.149(4)	O(1)#1–Mn(1)–N(4)	93.33(15)
Mn(1)–N(4)	2.233(5)	N(4)#1–Mn(1)–N(4)	103.3(2)
Mn(1)–N(3)	2.257(5)	O(1)–Mn(1)–N(3)	99.62(17)
N(1)#1–Cu(1)–N(1)	87.8(2)	O(1)#1–Mn(1)–N(3)	91.91(18)
N(1)#1–Cu(1)–N(2)	163.62(18)	N(4)#1–Mn(1)–N(3)	72.96(19)
N(1)–Cu(1)–N(2)	92.30(17)	N(4)–Mn(1)–N(3)	97.87(18)
N(1)–Cu(1)–N(2)#1	163.62(18)	O(1)–Mn(1)–N(3)#1	91.91(18)
N(2)–Cu(1)–N(2)#1	92.2(3)	N(4)–Mn(1)–N(3)#1	72.97(19)
O(1)–Mn(1)–O(1)#1	74.18(19)	N(3)–Mn(1)–N(3)#1	165.6(3)

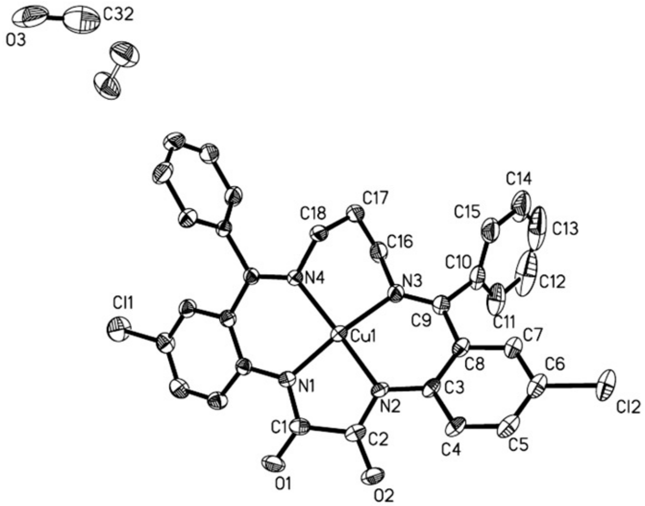

Figure 1. Perspective view of complex **1** with labelling scheme.

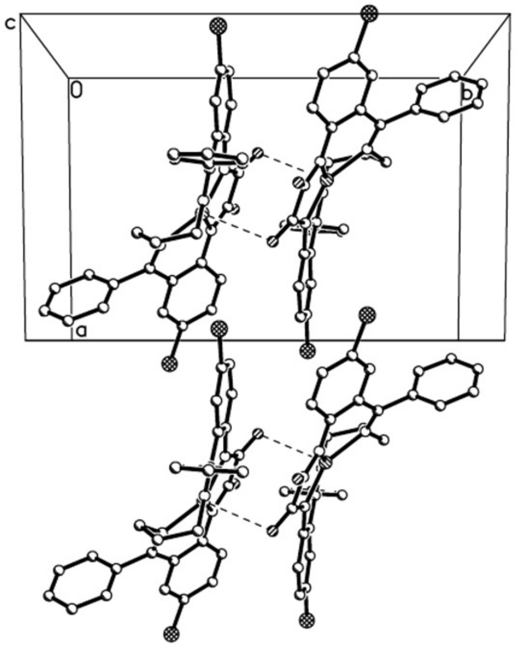

Figure 2. The packing of complex **1**. There is a weak coordination bond between Cu···O.

3.2. *Crystal structure of complex 2*

The results of X-ray diffraction show that the structure the complex **2** consist of $[Mn(bpy)_2(CuL)]^{2+}$; two H_2O molecules and two perchloric groups. Table 1 lists the crystal data and refinement details of complex **2**. Table 3 lists the part of bond lengths and bond angles of complex **2**. Figure 3 shows the structure of the $[Mn(bpy)_2(CuL)]^{2+}$

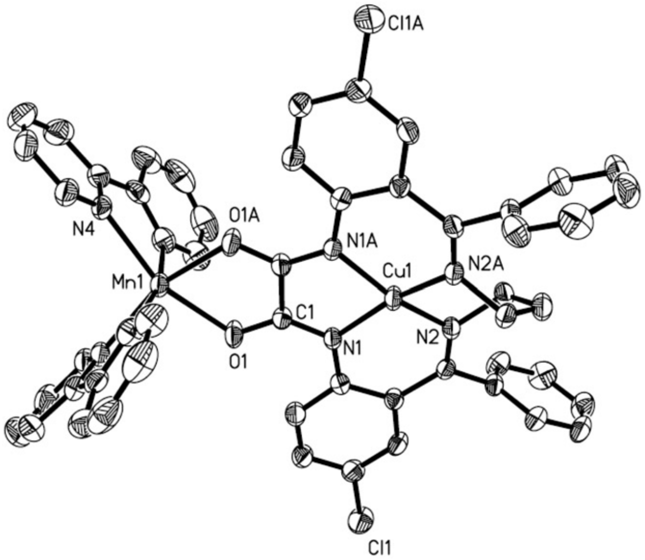

Figure 3. Perspective view of complex **2**. The two water molecules and two perchloric anions in the cell were omitted for clarity.

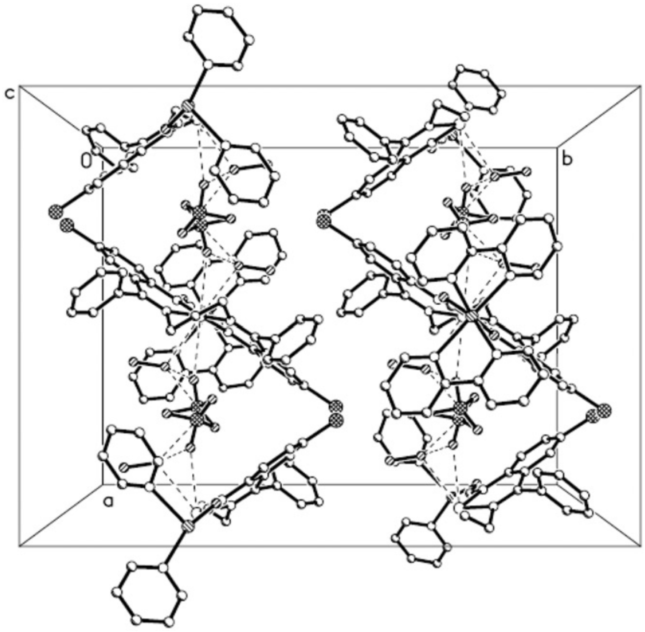

Figure 4. The packing diagram of complex **2**.

cation in the complex. The coordination environments of all the terminal Cu(II) ions are similar to the Cu(II) of CuL precursor. The Mn(II) centre is coordinated by four nitrogen atoms from two 2,2′-dipyridyl and two oxygen atoms from the Cu(II) precursor. Figure 4 shows the packing diagram of complex **2**. There are two weak interactions in the crystal which make the molecule form 3D framework. One is H-bond which lies in C–H $\cdots$ N(2.669 Å) and C–H $\cdots$ O(2.592 Å), the other is weak coordination link between Cu(II) and oxygen atoms from perchloric anions or water molecules (Cu $\cdots$ O(H_2O) is 2.848 Å, Cu $\cdots$ O(ClO_4^-) is 2.975 Å).

3.3. *Spectroscopic properties*

In complex **1**, the IR spectra show that there are two distinct band in 1670 and 1650 cm^{-1}, assigned to the two ν(C=O). 1610, 1590, and 1550 cm^{-1} are relative to ν(C=N) bands. The IR spectra of complex **2** showing three strong bands around 1640, 1604 and 1481 cm^{-1}, attributed to the ν(N–C–O) stretching bands, are characteristic of the bridging oxamide group [20]. The ν(C=O) bands of oxamido group of the binuclear complexes are seen around 1640 cm^{-1}, which are lower relative to that of mononuclear CuL(1670 cm^{-1}). The IR bands around 1570 cm^{-1} are assigned to the ν(C=N) mode of the macrocyclic ligand. The broad band around 1110 cm^{-1} is attributed to the perchlorate. The lack of any splitting of the perchlorate peak is consistent with noncoordinated tetrahedral ClO_4^- [21].

The electronic absorption spectrums of the two complexes in DMF are very similar. Both complexes display intense bands at 376 and 267 nm, due to intraligand and charge-transfer transitions in the [CuL] chromophore [22]. Moreover, a weak broad absorption at 625 nm for **1** (629 nm for **2**) can be assigned to the d–d transitions of copper atoms in tetrahedral coordination environments.

3.4. *Magnetic properties*

The magnetic behavior of complex **2** are investigated in the 80–300 K temperature range on a LDJ 9600 vibrating sample magnetometer in an applied magnetic field of 5000 G. The room temperature value for μ_{eff} is 5.01 μ_B, (where μ_{eff} is the effective magnetic moment), upon cooling to 80 K, the μ_{eff} value steadily decreases to 4.42 μ_B at 85 K, suggesting that an antiferromagnetic interaction between Cu(II)–Mn(II) is operative. The fit of the experimental data was performed according to the following Hamiltonian: $\hat{H} = -2J\hat{S}_{Cu}\hat{S}_{Mn}$

$$\chi_M = \frac{N\beta^2}{k(T-\theta)} \frac{28g_3^2 + 10g_2^2 \exp(-6J/kT)}{7 + 5\exp(-6J/kT)}$$

$$g_2 = \left(\frac{7g_{Mn} - g_{Cu}}{6}\right)$$

$$g_3 = \left(\frac{5g_{Mn} + g_{Cu}}{6}\right)$$

The least squares fitting of the experimental data led to $J = -74.1\ cm^{-1}$, $g_{Cu} = 2.04$, $g_{Mn} = 1.84$ and $R = 1.27 \times 10^{-3}$. The J values are negative, also indicating the antiferromagnetic interaction between the Cu(II) and Mn(II) ions.

Supplementary material

Full crystallographic data for the two complexes have been deposited with Cambridge Crystallographic Data centre, CCDC reference No. 265290 for **1** and

265291 for **2**, respectively. Copies of the data can be obtained, free of charge, from the CCDC, 12 Union Road, Cambridge CB21EZ, UK (Fax: +44-1223-336-033; Email: deposit@ccdc.cam.ac.uk).

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