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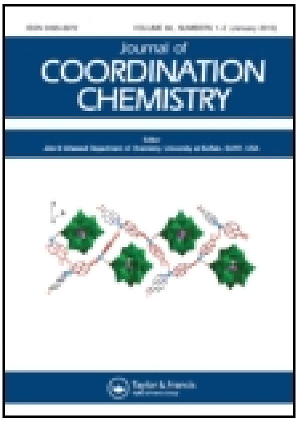

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Synthesis, crystal structure, and magnetic properties of a salt containing $[Cu_2Cl_7]^{3-}$ and 4-nitrobenzyl-4′-dimethylaminopyridinium

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Synthesis, crystal structure, and magnetic properties of a salt containing $[Cu_2Cl_7]^{3-}$ and 4-nitrobenzyl-4′-dimethylaminopyridinium

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A new salt, $[NO_2BzDMAP]_3[Cu_2Cl_7] \cdot H_2O$ (**1**), has been synthesized, where $[NO_2BzDMAP]^+$ is 1-(4′-nitrobenzyl)-4-dimethylaminopyridinium. Herein, the synthesis, spectral and structural characterization, and magnetic behavior of **1** are reported. It is orthorhombic, with space group $Pca2_1$, and $a = 26.639(2)$ Å, $b = 9.638(1)$ Å, and $c = 20.011(2)$ Å with $V = 5137.5(7)$ Å^3 for $Z = 4$. The anion shows a chloride-bridged binuclear structure with $Cu \cdots Cu$ distance of 3.872 Å; the two Cu(II) ions have a tetrahedral geometry. The cations stack through $p \cdots \pi$ and $\pi \cdots \pi$ interactions, and a complicated hydrogen-bonding network structure is formed through $C–H \cdots Cl$ and $C–H \cdots O$ hydrogen bonds. The variable temperature magnetic susceptibility measurements reveal that **1** exhibits strong antiferromagnetic interaction with $J = -193.0\,cm^{-1}$.

Keywords: Binuclear multichlorocuprate(II); 4-Nitrobenzyl-4′-dimethylaminopyridinium; Chloride-bridged; Crystal structure; Antiferromagnetic interaction

1. Introduction

The crystal chemistry of copper(II) halides has been extensively studied due to their structural variety, modes of coordination, and applications in chemical and material sciences [1–7]. The bridging capabilities of the halides and the electrostatic effects of organic counterions result in the 2-D layer perovskite structural class [8], stacked 1-D chains [9], and discrete dimers [10, 11]. Attention has been paid to copper(II) halide complexes, such as trigonal planar CuX_3 and tetrahedral CuX_4 geometries [12–16]. Especially, salts of the type A_2CuX_4 (A = cation, X = halide) have played an important role in development in low-dimensional magnetism due to their 2-D layer perovskite structure [6, 8]; the magnetic properties of this family are related to the interionic distances between neighboring $[CuX_4]^{2-}$ and the properties of the cations. For dimeric copper(II) halide complexes, the dominant species found are $[Cu_2X_6]^{2-}$ [17]. Especially, among complexes containing $[M_2X_7]^{3-}$ (where M = Hg, Co, Cu), there are numerous examples containing Hg [18–20], while only two containing Co have been

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reported [21, 22] and none with Cu. Derivatives of benzylpyridinium (abbreviated as $[RBzPy]^+$) are flexible and multifunctional cations containing two aromatic rings in which the molecular configuration was determined by the dihedral angles between these rings [23–25]. We have used this series of flexible cations as the counterion of $[CuX_4]^{2-}$ to develop a new class of salts [26, 27]. In this article, the first complex containing $[Cu_2X_7]^{3-}$, $[NO_2BzDMAP]_3[Cu_2Cl_7] \cdot H_2O$ (**1**), has been obtained by direct reaction of $[NO_2BzDMAP]Cl$ and $CuCl_2$ in ethanol acidified with concentrated hydrochloric acid; spectra, crystal structure, and magnetic behavior have been investigated.

2. Experimental

2.1. *General materials and physical measurements*

4-Nitrobenzyl chloride and 4-dimethylaminopyridine were purchased from Fluka Chemical Company and used without purification. $CuCl_2 \cdot 2H_2O$ and hydrochloric acid were obtained from Aldrich Chemical Company and used as received. 1-(4′-Nitrobenzyl)-4-dimethylaminopyridinium chloride ($[NO_2BzDMAP]Cl$) was prepared by the literature method [28]. Chemical compositions were analyzed by combustion analysis spectrum for C, N, and H on a Perkin-Elmer 240C Elemental Analyzer. IR spectra were collected on an IF66V FT–IR (400–4000 cm^{-1} region) spectrophotometer using KBr pellets of the samples. Electrospray mass spectra [ESI-MS] were determined on a Finnigan LCQ mass spectrograph, sample concentration $1.0 \times 10^{-5}\,mol\,L^{-1}$. Magnetic data were collected using a Quantum Design MPMS superconducting quantum interference device (SQUID) magnetometer. Crushed single crystals of the salt were packed into a small gelatin capsule and temperature-dependent magnetic susceptibility data were collected from 2 to 300 K at an applied magnetic field of 2 kOe. The diamagnetic data of the capsule without sample were determined to deduct the corresponding diamagnetic contribution and obtain the magnetic data of the sample. Diamagnetic correction was made with Pascal's constants for all constituent atoms and the temperature-independent paramagnetic correction for copper, $60 \times 10^{-6}\,emu\,mol^{-1}$, was applied to the data sets.

2.2. *Synthesis of tri[1-(4′-nitrobenzyl)-4-dimethylaminopyridinium][Cu_2Cl_7]•H_2O (1)*

$[NO_2BzDMAP]Cl$ (0.44 g, 1.5 mmol) and $CuCl_2 \cdot 2H_2O$ (0.17 g, 1 mmol) was dissolved in 20 mL of ethanol acidified with 3 mL of concentrated hydrochloric acid. The green solution was heated and stirred under reflux for 1 h. Brown crystals were developed by evaporation of the solution at room temperature after several days. The crystals were washed with diethyl ether after filtration. Yield: 0.43 g, 74%. Anal. Calcd for $C_{42}H_{48}N_9O_6Cu_2Cl_7 \cdot H_2O$ (%): C, 43.18; H, 4.31; N, 10.79. Found (%): C, 43.28; H, 4.46; N, 10.65.

2.3. *Determination of crystal structure*

Intensity data of **1** were collected using a SMART CCD area detector (Mo-Kα radiation, $\lambda = 0.71073$ Å) at 291 K. The structure was solved by direct methods using

Table 1. Crystal data and structure refinement summary for **1**.

Empirical formula	$C_{42}H_{48}N_9O_6Cu_2Cl_7 \cdot H_2O$
Formula weight	1168.14
Temperature (K)	291(2)
Crystal system	Orthorhombic
Space group	$Pca2_1$
Unit cell dimensions (Å)	
a	26.639(2)
b	9.638(1)
c	20.011(2)
Volume (Å^3), Z	5137.5(7), 4
Crystal size (mm^3)	$0.10 \times 0.12 \times 0.18$
D_{Calcd} (g cm^{-3})	1.510
μ (Mo-Kα) (mm^{-1})	1.248
F(000)	2392
2θ range (°)	3.06–50.00
Limiting indices	$-31 \leq h \leq 31$; $-11 \leq k \leq 11$; $-22 \leq l \leq 23$
Reflections collected/unique	35,525/8651
Independent reflection	7099
R_{int}	0.0394
Goodness-of-fit on F^2	1.014
Data/restraints/parameters	8651/1/610
R and wR [$I > 2\sigma(I)$]	0.0522, 0.1349
R and wR (all data)	0.0671, 0.1432
Largest difference peak and hole (e Å^{-3})	0.517 and −0.281

SHELXS 97 and refined on F^2 by full-matrix least squares (SHELXL 97) [29]. All non-hydrogen atoms were easily found from the Fourier map and refined anisotropically. Hydrogen atoms were constrained to ride on the respective carbons with an isotropic displacement parameter equal to 1.2 times the equivalent isotropic displacement parameter of their parent atom. The details of data collection, refinement, and crystallographic data of **1** are summarized in table 1. Selected bond lengths and angles for **1** are listed in table 2.

3. Results and discussion

3.1. *Syntheses, IR spectra, and ESI-MS*

Brown single crystals of $[NO_2BzDMAP]_3[Cu_2Cl_7] \cdot H_2O$ were grown by slow evaporation at room temperature from ethanol solutions. Elemental analyses are in agreement with expected values. In IR spectra of **1**, bands at 3046(m), 2970(m), and 2934(m) cm^{-1} for **1** are due to ν(C–H) of the aromatic rings and methylene. Bands at 1649(m), 1572(m), 1439(m), and 1403(m) cm^{-1} for **1** can be assigned to ν(C=N) and ν(C=C) of the pyridine and phenyl rings. Bands at 1522(s) and 1346(s) cm^{-1} are the ν(Ar–NO_2) bands for **1**. Positive-ion ESI-MS spectra of **1** in MeOH show that the mass spectrum is dominated by the 258.1 peak which is due to $[NO_2BzDMAP]^+$ ion.

3.2. *Crystal structure*

The molecular structure of $[NO_2BzDMAP]_3[Cu_2Cl_7] \cdot H_2O$ is shown in figure 1. Compound **1** crystallizes in the orthorhombic system with space group $Pca2_1$ and the

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

Cu(1)–Cl(1)	2.231(2)	N(2)–C(7)	1.484(10)
Cu(1)–Cl(2)	2.250(2)	N(2)–C(8)	1.344(10)
Cu(1)–Cl(3)	2.211(2)	N(2)–C(12)	1.347(9)
Cu(1)–Cl(4)	2.3048(19)	N(5)–C(21)	1.466(10)
Cu(2)–Cl(4)	2.322(2)	N(5)–C(22)	1.360(9)
Cu(2)–Cl(5)	2.231(2)	N(5)–C(26)	1.353(10)
Cu(2)–Cl(6)	2.252(2)	N(8)–C(35)	1.491(10)
Cu(2)–Cl(7)	2.2443(19)	N(8)–C(36)	1.348(9)
Cl(1)–Cu(1)–Cl(2)	95.29(8)	Cl(5)–Cu(2)–Cl(7)	139.05(8)
Cl(1)–Cu(1)–Cl(3)	97.74(8)	Cl(6)–Cu(2)–Cl(7)	97.29(8)
Cl(1)–Cu(1)–Cl(4)	151.11(7)	Cu(1)–Cl(4)–Cu(2)	113.63(7)
Cl(2)–Cu(1)–Cl(3)	145.77(8)	C(7)–N(2)–C(8)	122.0(7)
Cl(2)–Cu(1)–Cl(4)	90.17(7)	C(7)–N(2)–C(12)	119.8(6)
Cl(3)–Cu(1)–Cl(4)	93.39(8)	C(8)–N(2)–C(12)	118.2(7)
Cl(4)–Cu(2)–Cl(5)	90.58(7)	C(21)–N(5)–C(22)	120.3(6)
Cl(4)–Cu(2)–Cl(6)	150.60(7)	C(21)–N(5)–C(26)	121.5(6)
Cl(4)–Cu(2)–Cl(7)	94.20(7)	C(35)–N(8)–C(36)	120.2(6)
Cl(5)–Cu(2)–Cl(6)	98.12(8)	C(35)–N(8)–C(40)	120.3(6)

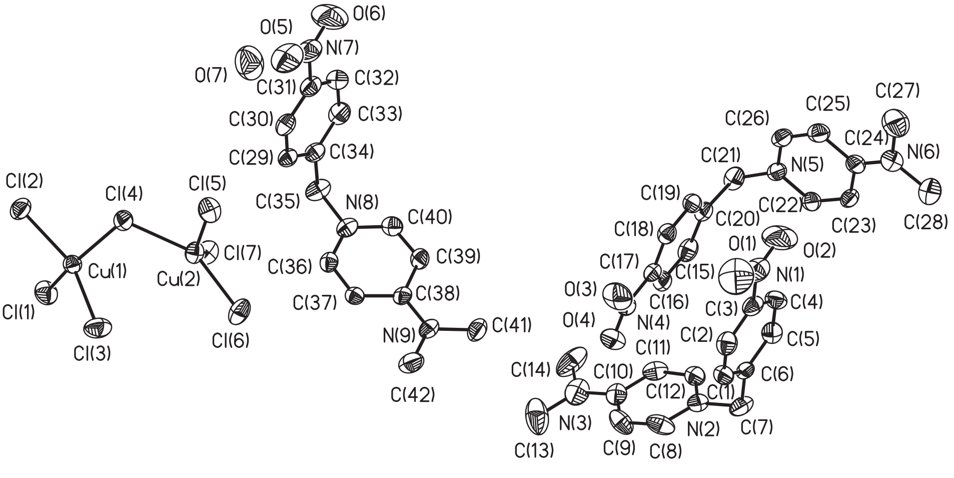

Figure 1. ORTEP plot (30% probability ellipsoids) of **1**; hydrogen atoms have been omitted for clarity.

asymmetric unit within the unit cell comprises one $[Cu_2Cl_7]^{3-}$, three $[NO_2BzDMAP]^+$, and one water molecule. The anion shows a chloride-bridged binuclear structure, and both Cu(II) ions are coordinated by three terminal and a bridging chloride (figure 2a). The Cu ··· Cu distance of 3.872(2) Å is non-bonding. The mean bond distance of Cu–Cl (bridging) is 2.313(2) Å, larger than the Cu–Cl (terminal) distance: mean 2.236(2) Å. The average angle of Cl–Cu–Cl was 111.94(8)°, while Cu(1)–Cl(4)–Cu(2) is 113.63(7)°, these values are in agreement with those of $[CuCl_4]^{2-}$ salts previously reported [4, 7]. Three $[NO_2BzDMAP]^+$ cations adopt the conformation where both the phenyl and pyridine rings are twisted to the C–C–N reference plane. For the cation containing N(2), the dihedral angle which the phenyl/pyridine ring makes with the reference plane of C(6)–C(7)–N(2) is 78.2(2)° (θ_1)/91.1(2)° (θ_2), and two aromatic rings make a dihedral

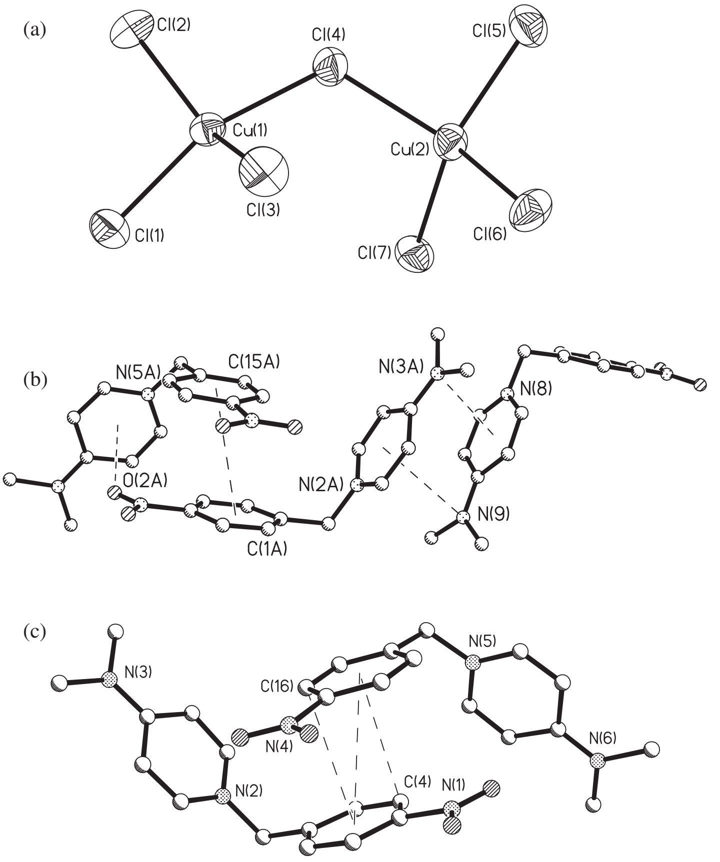

Figure 2. (a) The structure of $[Cu_2Cl_7]^{3-}$. (b) and (c) The $p\cdots\pi$ and $\pi\cdots\pi$ stacking interactions between adjacent $[NO_2BzDMAP]^+$ cations.

angle of 69.5(2)° (θ_3). For cation containing N(5), these dihedral angles θ_1, θ_2, and θ_3 are 56.4(2)°, 94.5(2)°, and 81.7(2)°, respectively. For the cation containing N(8), these dihedral angles θ_1, θ_2, and θ_3 are 103.2(2)°, 91.1(2)°, and 106.7(2)°, respectively. Three kinds of weak interactions are observed between adjacent cations: (1) the $p\cdots\pi$ interactions [30, 31] between O(2) of the NO_2 and the pyridine rings containing N(5) with distance of 3.744(1) Å from O(2) to the centroid of the ring (figure 2b); (2) the $\pi\cdots\pi$ stacking interactions between the C(1 A)–C(6A) and the C(15A)–C(20A) phenyl rings, as shown in figure 2(c); the distances between C(4) and C(16) to the centroids of the rings are 3.514(1) and 3.563(1) Å with slip angles of 21.8(2)° and 21.5(2)°; (3) the $p\cdots\pi$ interactions (figure 2b) between nitrogen atoms and adjacent pyridine rings, and the distance of the N(3) and N(9) to the rings are 3.425(1) Å and 3.658(1) Å, respectively. Stacking of the cations is shown in figure 3(a). In addition, there are six weak C–H···Cl hydrogen bonds [32, 33] and three weak C–H$\cdots$O hydrogen bonds [33] observed between adjacent anions and cations, the bond parameters are listed in table 3. These interactions in the crystal result in a complicated hydrogen-bonding network structure (figure 3b).

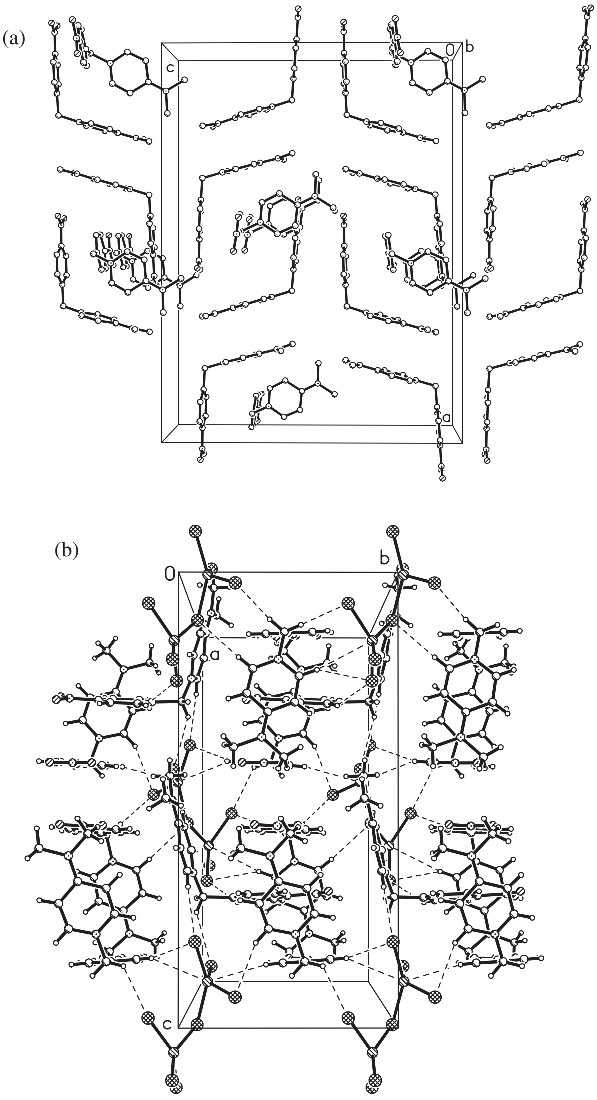

Figure 3. (a) The 2-D structure of adjacent cations by $p\cdots\pi$ and $\pi\cdots\pi$ stacking interactions. (b) The hydrogen-bonding network structure of **1** viewing along the *a*-axis.

In the family of copper(II) chloride salts containing substituted pyridinium cations, the crystal stacking structure of the copper(II) chlorides is affected by the size and topology of the countercation and dominated by weak interactions such as $p\cdots\pi$, $\pi\cdots\pi$ interactions and hydrogen bonds. When the cation is 3-amino-2-chloropyridinium, a salt containing $[CuCl_4]^{2-}$ formed in which the anions form a linear chain through weak $Cl\cdots Cl$ interaction with a distance of 4.595 Å [34]; when 2-amino-3-chloro-5-trifluoromethylpyridinium is used, the anions create a dimeric structure containing $[Cu_2Cl_8]^{4-}$ *via* bridging Cl^-, and these dimers further form ladders through short $Cl\cdots Cl$ contacts [35]. When 1-(4′-nitrobenzyl)-4-dimethylaminopyridinium is

Table 3. Weak hydrogen bonds for **1** (Å and °).

D–H···A	*d*(D–H)	*d*(H···A)	*d*(D···A)	∠(DHA)
C(7)–H(7B)···Cl(7) #1	0.970	2.730	3.676(9)	164.00
C(14)–H(14B)···O(5) #2	0.960	2.500	3.275(10)	137.00
C(15)–H(15)···Cl(2) #3	0.930	2.780	3.591(7)	147.00
C(21)–H(21B)···Cl(5) #2	0.970	2.810	3.538(8)	132.00
C(25)–H(25)···Cl(1) #4	0.930	2.820	3.696(8)	157.00
C(27)–H(27B)···O(7) #3	0.960	2.570	3.519(15)	172.00
C(35)–H(35B)···Cl(3) #5	0.970	2.780	3.459(8)	128.00
C(36)–H(36)···Cl(7)	0.930	2.790	3.659(6)	156.00
C(41)–H(41C)···O(1) #6	0.960	2.430	3.294(11)	150.00

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

utilized, the first complex containing $[Cu_2X_7]^{3-}$ was obtained in which the cations stack through $p\cdots\pi$ and $\pi\cdots\pi$ interactions, and a complicated hydrogen-bonding network structure is formed through C–H···Cl and C–H···O hydrogen bonds.

3.3. *Magnetic properties*

Variable temperature magnetic susceptibility measurements were carried out on a polycrystalline **1** under an applied field of 2000 Oe from 2 to 300 K. The $\chi_M T$ and χ_M *versus* T plots are shown in figure 4(a) and (b). The room temperature (300 K) $\chi_M T$ is 0.634 emu $\mathrm{K\,mol^{-1}}$, distinctly lower than that expected for two uncoupled $S = 1/2$ Cu(II) spins with $g = 2.0$. As the temperature is lowered, the value of $\chi_M T$ decreases rapidly to 0.0536 emu $\mathrm{K\,mol^{-1}}$ at 50 K, then decreases slowly to 0.0299 emu $\mathrm{K\,mol^{-1}}$. This behavior is a signature of strong antiferromagnetic interaction. The experimental data have been fitted using the dinuclear model formula (1), where the Hamiltonian $H = -2JS_AS_B$, $S_A = S_B = 1/2$ [36, 37],

$$\chi_M = (2N\beta^2g^2/kT)(1-\rho)/(3+\exp(-2J/kT)) + (N\beta^2g^2/2kT)\rho, \qquad (1)$$

where N, g, k, and β have their usual meanings [28], ρ is the percentage of the paramagnetic impurities effect on magnetism, and J is the exchange coupling parameter describing the magnetic interaction between any two neighboring $S = 1/2$ spins. The best-fit parameters obtained by least-squares fit are: $g = 2.11$, $J = -193.0\,\mathrm{cm^{-1}}$, $\rho = 3.8\times10^{-2}$, and $R = 2.5\times10^{-6}$ (R is defined as $\Sigma\ (\chi_M^{\mathrm{calcd}} - \chi_M^{\mathrm{obsd}})^2/(\chi_M^{\mathrm{obsd}})^2$). The predicted curve is the red solid line in figure 4(b), agreement between the experimental data and the theoretical curve is excellent as indicated by the low value of R. $[NO_2BzDMAP]_3[Cu_2Cl_7]$ is different from *bis*(3-amino-2-chloropyridinium) tetrachlorocuprate(II) [34] and *bis*(2-amino-3-chloro-5-trifluoromethylpyridinium) tetrachlorocuprate(II) [35] in its magnetic properties, which can be attributed to the crystal structures.

4. Conclusion

The synthesis, spectral and structural characterization, and magnetic behavior of a new salt $[NO_2BzDMAP]_3[Cu_2Cl_7]\cdot H_2O$ (**1**) has been reported. The $[Cu_2Cl_7]^{3-}$ shows a

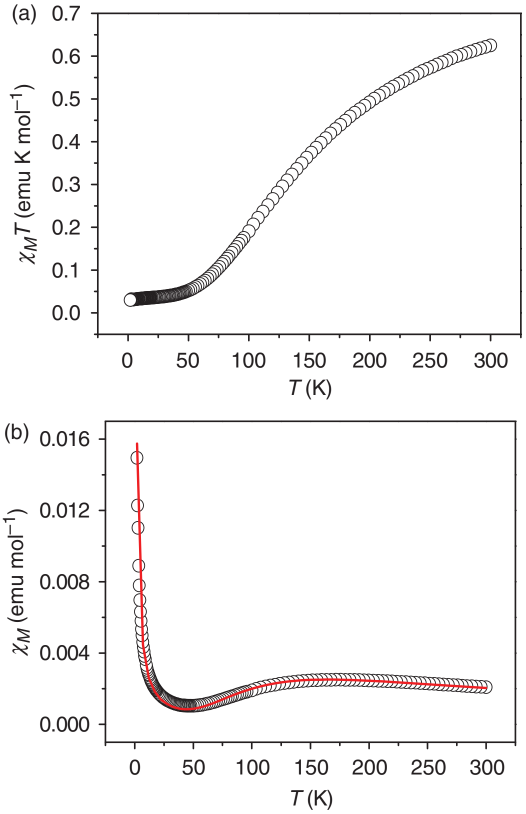

Figure 4. (a) Plot of $\chi_M T$ *vs.* T for **1**. (b) Plot of χ_M *vs.* T. The red solid line is from the theoretic calculations and detailed fitting procedure described in the text.

chloride-bridged binuclear structure with Cu $\cdots$ Cu distance of 3.872 Å. The two Cu(II) ions exhibit tetrahedral geometry. The cations stack through $p \cdots \pi$ and $\pi \cdots \pi$ interactions and C–H $\cdots$ Cl and C–H $\cdots$ O hydrogen bonds result in a complicated hydrogen-bonding network structure. Variable temperature magnetic susceptibility measurements reveal that **1** exhibits a strong antiferromagnetic interaction with $J = -193.0\,\mathrm{cm}^{-1}$.

Supplementary material

The detailed crystallographic data of **1** have been deposited at the Cambridge Crystallographic Data Center as supplementary publication No. CCDC-837448. Copies of the data may be obtained free of charge from the Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (Fax: +44-1223-336033; E-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

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