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Polynuclear complexes incorporating Cu(II) and Mn(II) centers bridged by acetylenedicarboxylate: Structure, thermal stability and magnetism

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Abstract

Two acetylenedicarboxylate complexes, $[Mn_2(C_4O_4)(phen)_4(H_2O)_2](ClO_4)_2 \cdot H_2O$ (1) and $[Cu(C_4O_4)(2,2'-bpy)]_n$ (2), have been synthesized and characterized by elemental analyses and IR spectra. Mn^{II} ions have distorted octahedral coordination geometry and bridged by $C_4O_4^{2-}$ to form a binuclear structure. Thermal analysis shows $C_4O_4^{2-}$ released in the range of 180–280 °C. Cu^{II} is five-coordinated by 2,2'-bipyridine and three oxygen atoms from three different $C_4O_4^{2-}$ ligands, leading to a square-pyramidal coordination geometry. Each $C_4O_4^{2-}$ links three Cu^{II} centers through bifunctional carboxylate to form a polymeric structure of (4, 4) layers with nodes being dimer of Cu_2O_2 and bridges of $C_4O_4^{2-}$. Its variable-temperature magnetism was also investigated. © 2006 Elsevier B.V. All rights reserved.

Keywords: Copper; Manganese; Acetylenedicarboxylate; Crystal structure; Thermal analysis; Magnetism

1. Introduction

Coordination polymers are of great interest due to their intriguing structural motifs and potential applications in optical, electronic, magnetic, and porous materials [1–3]. Many of them are based on metal carboxylates [4], such as dicarboxylate succinate, maleate and terephthalate [5–9], polycarboxylate 1,3,5-benzenetricarboxylate and 1,2,4,5-benzenetetracarboxylate complexes [10,11]. Analogous to rigid aromatic dicarboxylate terephthalate, acetylenedicarboxylate ($C_4O_4^{2-}$) is a linear dicarboxylate dianion with shorter bridge and acetylene replacing aromatic group, which is a potential worthy bridging ligand in constructing coordination polymer. Up to now, only a few acetylenedicarboxylate complexes were reported, such as linear complexes [$C_0(C_4O_4)(H_2O)_4]_n$ and $[Cd(C_4O_4) (H_2O)_3]_n [12-14]$, metal-organic framework $[Zn(C_4O_4)_2 (HTEA)_2]_n [15]$, rare-earth complexes $[Eu_2(C_4O_4) (CO_3)_2(H_2O)_2]$ and $[Ln_2(C_4O_4)_3(H_2O)_6] [16,17]$. We present here two new acetylenedicarboxylate complexes $[Mn_2(C_4O_4)(phen)_4(H_2O)_2] (ClO_4)_2 \cdot H_2O (1)$ and $[Cu(C_4O_4) (2,2'-bpy)]_n (2)$. Complex 1 is dimer in which two Mn(II) ions are bridged by $C_4O_4^{2-}$, while complex 2 is a polymeric complex of (4,4) layers with nodes being dimer of Cu_2O_2 and bridges of $C_4O_4^{2-}$. Their IR spectra, thermal stability and magnetism are also reported.

2. Experimental

2.1. Materials and physical measurement

All chemicals were purchased from commercial sources and were used as received without further purification. Elemental analyses were performed on a Perkin-Elmer 240C analyzer. Infrared spectra were recorded on a Nicolet A370 FT-IR spectrometer in the 4000–400 cm⁻¹ region

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Table 1 Crystal data and structure refinement for complex 1 and 2

	1	2
Empirical formula	$C_{52}H_{38}Mn_2Cl_2N_8O_{15}$	C14H8CuN2O4
Formula weight	1195.68	331.76
Crystal system	Triclinic	Monoclinic
Space group	$P\overline{1}$	P2(1)/c
<i>a</i> (Å)	11.880(4)	9.842(2)
b (Å)	13.975(4)	14.782(3)
c (Å)	17.188(5)	8.549(2)
α (°)	104.342(4)	90
β (°)	106.854(3)	102.34(3)
γ (°)	96.405(4)	90
$V(\text{\AA}^3)$	2593.6(14)	1215.0(4)
Ζ	2	4
$D_{\rm c} ({\rm g \cdot cm^{-3}})$	1.531	1.814
$\mu (\mathrm{mm}^{-1})$	0.668	1.816
Crystal size (mm ³)	$0.10 \times 0.10 \times 0.02$	$0.20 \times 0.18 \times 0.16$
$T(\mathbf{K})$	293(2)	291(2)
F_{000}	1220	668
Reflections collected	10845	4195
Unique reflections	8982 $[R_{\rm int} = 0.0174]$	2468 $[R_{int} = 0.0587]$
Data/restraints/parameters	8982/10/756	2468/0/191
$R[I \ge 2\sigma(I)]$	$R_1 = 0.0605, wR_2 = 0.1445$	$R_1 = 0.0502, wR_2 = 0.1199$
R (all data)	$R_1 = 0.0986, wR_2 = 0.1730$	$R_1 = 0.0734, wR_2 = 0.1294$
Goodness-of fit on F^2	1.032	1.123
Largest diff. peak and hole	0.855 and $-0.417 \text{ e} \cdot \text{\AA}^{-3}$	$0.654 \text{ and } -0.775 \text{ e} \text{-} \text{\AA}^{-3}$

using KBr disks. TG-DSC analysis was carried out on a Netzsch STA 449C thermal instrument at a heating rate of 10 °Cmin⁻¹ in the atmosphere. Magnetic susceptibilities (2–300 K) were measured using a Quantum Design PPMS-9 magnetometer and diamagnetic correction was made with Pascal's constants.

2.2. Synthesis

 $[Mn_2(C_4O_4)(phen)_4(H_2O)_2](ClO_4)_2 H_2O$ (1). Five milliliters of aqueous solution containing acetylenedicarboxylic acid (0.029 g, 0.25 mmol), piperidine (0.043 g, 0.50 mmol) was added to 5 mL aqueous solution of $Mn(ClO_4)_2$ ·6H₂O (0.181 g, 0.50 mmol) under stirring. To this mixture was added 5 mL methanol solution of 1,10-phenanthroline (0.180 g, 1.0 mmol). The resulting solution was left undisturbed at room temperature for 2 weeks. Light yellow crystals 1 were harvested, washed with cool water and dried in air. The yield is 45% based on Mn. IR (cm^{-1} , KBr): 3431(w), 3215(w), 3059(w), 1623(s), 1590(s), 1517(m), 1425(m), 1323(s), 1103(vs), 846(m), 728(m), 623(w). Anal. calcd. for C₅₂H₃₈Mn₂Cl₂N₈O₁₅: C, 52.24; H, 3.20; N, 9.37. Found: C, 51.92; H, 3.22; N, 9.31. CAUTION! Perchlorate complexes of metal ions in the presence of organic ligands are potentially explosive. Only a small amount of material should be handled with care.

 $[Cu(C_4O_4)(2,2'-bpy)]_n$ (2). Five milliliters of aqueous solution containing acetylenedicarboxylic acid (0.057 g, 0.50 mmol) and piperidine (0.085 g, 1.0 mmol) was added to 5 mL aqueous solution of Cu(NO₃)₂·3H₂O (0.121 g, 0.50 mmol) under stirring. To this mixture was added 0.078 g (0.50 mmol) 2,2'-bipyridine dissolved in 5 mL

methanol solution. The resulting solution was left undisturbed at room temperature for 3 weeks. Blue crystals **2** were harvested, washed with cool water and dried in air. The yield is 73% based on Cu. IR (cm⁻¹, KBr): 3060(w), 1641(s), 1620(s), 1600(m), 1474(w), 1444(m), 1333(s), 1295(s), 1153(w), 1004(w), 764(m), 728(w), 687(m). Anal. calcd. for $C_{14}H_8CuN_2O_4$: C, 50.68; H, 2.43; N, 8.44. Found: C, 51.12; H, 2.41; N, 8.39.

2.3. X-ray crystallography

Data collection for **1** was performed on a Bruker Smart Apex CCD diffractometer and for **2** on a Rigaku RAXIS-IV IP diffractometer, using graphite-monochromatic Mo K α radiation ($\lambda = 0.71073$ Å). Determinations of the crystal system, orientation matrix and cell dimensions were performed according to the established procedures. Lorentz polarization and absorption correction were applied. The structures were solved by direct method and refined by full-matrix least-squares on F^2 using SHELX-97 program package [18]. All nonhydrogen atoms were refined anisotropically, and hydrogen atoms were added geometrically. The crystal data and structure refinement results are summarized in Table 1.

3. Results and discussion

3.1. Synthesis and IR spectra

Acetylenedicarboxylic acid mixed with piperidine to yield $C_4 O_4^{2-}$ dianion, here piperidine acted as an

organic base. Piperidine salt of $C_4 O_4^{\ 2-}$ reacted with $Mn(ClO_4)_2 \cdot 6H_2O$ and phen to produce a binuclear manganese complex 1. IR spectrum indicated 1 possesses $C_4O_4^{2-}$, phen and water as ligands. Absorption peaks at 1623 and 1323 cm⁻¹ are assigned to $v_{as}(COO^{-})$ and $v_{s}(COO^{-})$ stretching vibrations of coordinated carboxylic groups, respectively. A 300 cm^{-1} separation between $v_{as}(COO^{-})$ and $v_{s}(COO^{-})$ is significantly larger than the 200 cm⁻¹ value of free carboxylate, indicating that the carboxylic groups of $C_4 O_4^{2-}$ coordinate to Mn(II) in a monodentate mode [19]. The characteristic strong absorption of ClO₄⁻ appears at 1103 cm^{-1} . Broad bands at ca. 3431 and 3215 cm^{-1} are attributed to the vibration absorptions of lattice water and coordinated water, respectively. Elemental analysis confirmed the formula $[Mn_2(C_4O_4)($ phen)₄ $(H_2O)_2$ (ClO₄)₂·H₂O.

When piperidine salt of $C_4O_4^{2-}$ reacts with $Cu(NO_3)_2$ ·3H₂O and 2,2'-bipy, a polymeric complex **2** was prepared. IR spectrum and elemental analysis suggested **2** has formula $[Cu(C_4O_4)(2,2'-\text{ bpy})]_n$. Complex **2** shows two $v_{as}(COO^-)$ absorption peaks at 1641, 1620 cm⁻¹ and

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

	8 () 8	() 1	
Mn(1)-O(1)	2.160(3)	Mn(2)–O(3)	2.112(9)
Mn(1) - O(5)	2.192(4)	Mn(2)–O(6)	2.146(4)
Mn(1) - N(1)	2.247(4)	Mn(2) - N(5)	2.259(4)
Mn(1) - N(2)	2.254(4)	Mn(2) - N(6)	2.313(3)
Mn(1)-N(3)	2.275(4)	Mn(2) - N(7)	2.277(4)
Mn(1) - N(4)	2.268(4)	Mn(2)–N(8)	2.273(4)
O(1) - Mn(1) - O(5)) 87.89(13)	O(3)–Mn(2)–O(6)	84.7(3)
O(1) - Mn(1) - N(1)) 99.82(13)	O(3)-Mn(2)-N(7)	100.9(3)
O(1) - Mn(1) - N(2)	88.09(13)	O(3)-Mn(2)-N(8)	102.7(3)
O(1) - Mn(1) - N(3)) 90.68(15)	O(3)-Mn(2)-N(5)	95.6(3)
O(1) - Mn(1) - N(4)) 163.26(15)	O(3)-Mn(2)-N(6)	167.5(3)
O(5) - Mn(1) - N(2)	168.69(16)	O(6)-Mn(2)-N(7)	165.98(15)
N(1)-Mn(1)-N(3)	6) 163.15(15)	N(5)-Mn(2)-N(8)	158.96(13)
N(1)-Mn(1)-N(2)	2) 73.95(16)	N(5)-Mn(2)-N(6)	72.77(13)
N(3)-Mn(1)-N(4)) 73.35(17)	N(7)-Mn(2)-N(8)	73.33(13)

two $v_s(COO^-)$ absorptions at 1333, 1295 cm⁻¹, which indicated that the carboxylic groups of C₄O₄²⁻ coordinate to Cu(II) in different modes. Crystal structure analysis further revealed that the carboxylic groups coordinate to Cu(II) in two modes.



Fig. 1. Molecular structure and packing diagram of complex 1 (perchlorate anions were omitted for clarity).

3.2. Structures description

 $[Mn_2(C_4O_4)(phen)_4(H_2O)_2](ClO_4)_2 \cdot H_2O$ 1 is a binuclear complex. Its molecular structure is shown in Fig. 1, while selected bond lengths and angles are listed in Table 2.

Acetylenedicarboxylate acts as bridging ligand, which is bound on each side to two Mn(II) ions through the monodentate carboxylate coordination mode. Mn(II) is six-coordinated and possesses a N₄O₂ coordination environment by one carboxylate O atom, four phen N atoms and one water molecule. Mn(1)-O(1) bond length is 2.160(3) Å, and Mn(1)-O(5) is 2.192(4) Å. Mn(1)-N average length is 2.261 Å. The bond lengths are in agreement with those in the structure of $[Mn_2(terephthalate)(phen)_4(H_2O)_2]_n$ $(ClO_4)_{2n}$ [20]. Larger deviations of bond angles from 90° of the idealized octahedral geometry, N(1)-Mn(1)- $N(2) = 73.95(16)^{\circ}$ and $N(3)-Mn(1)-N(4) = 73.35(17)^{\circ}$, indicate Mn(1) has a distorted octahedral coordination sphere. The basal plane is formed by atoms N(1), N(2), N(3), O(5), and the axial sites are occupied by atoms O(1), N(4). Two phen are in *cis* position around Mn(1), and their planes are nearly perpendicular to each other. $C_4O_4^{2-}$ links $[Mn(phen)_2(H_2O)]^+$ units to form a dimer. $Mn(1) \cdots Mn(2)$ separation is 9.874(2) Å. In addition, there exist remarkable π - π stacking interactions between adjacent phen ligands, while perchlorate anions act as counter ions appear in the crystal lattice.

 $[Cu(C_4O_4)(bipy)]_n$ **2** is a polymeric complex. Its molecular structure and packing diagram are illustrated in Fig. 2, while selected bond lengths and angles are listed in Table 3.

Cu(1) is five-coordinated by two 2,2'-bipy N atoms and three O atoms belonging to three different acetylenedicarb-

Table 3	
Selected bond lengths (Å) and angles (°) for complex 2	

Servered conta tengens (1) and angles (1) for compton 2					
Cu(1)–O(1)	1.931(3)	C(11)–O(1)	1.285(5)		
Cu(1)–O(4B)	1.978(3)	C(11)–O(2)	1.227(5)		
Cu(1)-O(4A)	2.350(3)	C(14)–O(3)	1.224(5)		
Cu(1)–N(1)	2.013(3)	C(14)–O(4)	1.277(5)		
Cu(1)–N(2)	2.008(3)	C(12)-C(13)	1.197(6)		
O(1)-Cu(1)-O(4B)	90.42(12)	O(1)-Cu(1)-N(2)	97.00(13)		
N(2)-Cu(1)-O(4B)	172.52(13)	O(1)-Cu(1)-N(1)	172.87(13)		
N(1)-Cu(1)-O(4B)	91.07(13)	O(1)-Cu(1)-O(4A)	94.80(12)		
N(2)-Cu(1)-O(4A)	100.15(11)	O(4A)-Cu(1)-O(4B)	78.20(12)		
N(1)-Cu(1)-O(4A)	92.33(12)	C(11)-C(12)-C(13)	175.5(4)		
Cu(1)-O(4A)-Cu(1C)	101.80(12)	C(12)-C(13)-C(14)	171.7(4)		

oxylate dianions with Cu(1)–O(1) bond length of 1.931(3) Å, Cu(1)–O(4A) 2.350(3) Å, and Cu(1)–O(4B) 1.978(3) Å. Cu(1)–O(4A) bond length is obviously longer than others. The coordination structure can be best described as a square-pyramidal geometry. The basal plane is formed by atoms O(1), N(1), N(2), O(4B), and the axial site is occupied by O(4A). Acetylenedicarboxylate is almost linear, while two carboxylic groups are non-coplanar with a torsion angle of 70.25°. Cu(1)···Cu(1A) separation is 7.58 Å, and Cu(1)···Cu(1C) separation is 3.37 Å. The coordination basal plane of Cu(1) is parallel to the plane of Cu(1C).

It is interesting to note that each $C_4O_4^{2-}$ ligand links three copper centers through two terminal carboxylate groups. One carboxylate acts as monodentate mode, while the other carboxylate exhibits an unusual bidentate bridging mode, linking two Cu^{II} centers using O atom to form a dimer of Cu₂O₂ (Cu–O–Cu angle 101.80°), which is rarely



Fig. 2. Molecular structure of complex 2.



Fig. 3. TG-DSC thermal analysis curves of complex 1.

observed in previous reports [21]. The structure of **2** is practically a complicated two-dimensional (4,4) network with nodes of Cu₂O₂ and bridges of C₄O₄²⁻.

3.3. Thermal stability and magnetism

TG-DSC analysis showed the thermal decomposition procedure of 1 as Fig. 3. The first endothermic process occurred in the temperature range of 80-130 °C, which corresponds to the release of lattice water and coordinated water (found 4.57%, calcd. 4.52%). The second weight-loss step appeared in the range of 180-280 °C with an exothermic peak at 209 °C, which corresponds to the decomposition of $C_4 O_4^{2-}$ (found 7.28%, calcd. 8.03%). This rather low decomposed temperature indicated that the acetylenedicarboxylate complex is thermal unstable [17]. McCormick has reported that linear chain complex $[Cu(C_4O_4)(H_2O_3)]$ was unstable at room temperature and decomposed explosively when heated [22]. The following weight-loss step indicated that one phen per unit released in the range $[Mn(phen)_2(H_2O)]$ of 300-420 °C (found 31.96%, calcd. 30.14%). Another phen may be lost later, following by a strong exothermic process of ClO_4^- decomposition. These processes occurred in the range of 430-680 °C, and overlapped in the TG curve with a total weight-loss of 42.49% (calcd. 42.77%). The final, thermal stable brown residue is MnO₂ (found 12.27%, calcd. 14.54%).

Variable-temperature magnetic susceptibilities of **2** have been measured in the range of 2–300 K (Fig. 4). The data can be best fitted with Curie-Weiss law, $\chi_m^{-1} = T/(C - \theta)$ with a Weiss constant $\theta = -1.5$ K. The effective magnetic moment (μ_{eff}) per Cu(II) unit at 300 K is 1.70 B.M., which is close to the spin-only value of free Cu²⁺ ion (S = 1/2, 1.73 B.M.). When temperature is lowered, μ_{eff} keeps no change between 11 and 300 K, and then decreases slowly to 1.56 B.M. at 2.02 K. These results indicate a very weak antiferromagnetic interaction in complex **2**, especially in the dimer of Cu₂O₂.

Magneto-structural correlation of carboxylate polynuclear complexes has been widely studied [23]. Although acetylenedicarboxylate is a worthy bridging ligand in constructing coordination polymer, we consider this bridge is still too long and the conjugation effect between carboxylic groups and ethynyl group is too weak to propagate magnetic interaction [20]. Usually, Cu₂O₂ dimer shows obvious magnetic couple. Most of oxygen- or carboxylate-bridging Cu^{II} dimers exhibit stronger antiferromagnetic couple [24]. However, the copper complex **2** has poor magnetic interaction. Further analysis shows that the Cu^{II} magnetic orbital $(d_{x^2-y^2})$ defines the unpaired electron lying on basal plane of square-pyramidal coordination geometry. Poor overlap between the two parallel magnetic orbital in the Cu₂O₂ dimer leads to the lack of magnetic interaction.

4. Conclusion

Two novel polynuclear complexes were prepared using acetylenedicarboxylate as bridging ligand. The binuclear manganese complex is stable up to 180 °C. The copper complex has polymeric structure, where each acetylenedicarboxylate bridges three copper centers to form two-dimensional (4,4) network with nodes being dimer of Cu_2O_2 and bridges of $C_4O_4^{2-}$. Poor overlap between the two parallel magnetic orbital in the Cu_2O_2 dimer leads to the lack of magnetic interaction.



Fig. 4. Temperature-dependent magnetic susceptibilities of complex 2.

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Appendix A. Supplementary data

Crystallographic data for the structural analyses have been deposited with the Cambridge Crystallographic Center, CCDC-281904 for Complex 1 and CCDC-251792 for complex 2. These data can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Center, 12, Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033). Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.molstruc. 2006.06.021.

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