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A novel heterohexanuclear Schiff base complex: Synthesis, structure and magnetic properties

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1. Introduction

The design and synthesis of polynuclear coordination compounds with predictable magnetic properties have always attracted the attention of inorganic chemists [1-4]. The selection of the ligand is the key to prepare polynuclear coordination complexes. The ligand disposition around the paramagnetic metal ions and the nature of the bridging atoms/groups influence these magnetic exchange interactions. In view of the above, the preparation of complexes with new chelating ligands to create new homoand heteronuclear magnetic materials is a challenge. Although great progress has been achieved in the synthesis homometallic coordination polymers, the polymeric complexes that bear two different metals are less common [3-6]. Schiff bases as good polydentate ligands have been widely used to synthesize homo/ heteropolynuclear complexes [7-15]. Although numerous di- and tri-nuclear Schiff base complexes have been reported [16-19]. However, to the best of our knowledge, any reports of Schiff base heterometallic hexanuclear complexes remains rare [20-26]. Taking into account of this, a new ligand with different types of Lewis donors, namely, N-N'-bis[(3-carboxylsalicylidene)]-N"-(2-aminoethyl)-1,2-ethanediamine (H₄L¹), was used in this study to build 3d-3d heterometallic coordination polymers. The main novelty of this work is that one imine bond in the ligand H_4L^1 is degraded into NH_2 group, giving the new ligand H_2L^2 (Scheme 1).

Herein, we report the syntheses, crystal structure and properties of a novel 3d–3d heterometallic hexanuclear Schiff base com-

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ABSTRACT

A novel heterohexanuclear Schiff base complex, $\{[CuMnL^1][CuL^2]\}_2 \cdot 2CH_3OH$ (1) $(H_4L^1 = N-N'-bis](3-carboxylsalicylidene)]-N''-(2-aminoethyl)-1,2-ethanediamine and <math>H_2L^2 = N-(3-carboxylsalicylidene)-N''-(2-aminoethyl)-1,2-ethanediamine)$ has been synthesized and characterized by elemental analysis, IR spectrum, thermogravimetric analysis (TGA), single-crystal X-ray diffraction and magnetic properties. Single-crystal structural analysis shows that complex 1 displays a rare heterohexanuclear structure. The asymmetry unit of complex 1 contains a trinuclear $[CuMnL^1][CuL^2]$ moieties. The result reveals that one imine bond in the ligand H_4L^1 is degraded into NH₂ group and that one 3-carboxylsalicylidene is thrown away, giving the new ligand H_2L^2 . Its magnetic properties have also been investigated. The interactions between metal ions in the trinuclear moiety are all antiferromagnetic.

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plex, namely, $\{[CuMnL^1][CuL^2]\}_2 \cdot 2CH_3OH$ (1). The complex was characterized by IR, elemental, X-ray crystallographic, and thermogravimetric analyses. The variable-temperature magnetic susceptibility behavior of 1 has been also studied.

2. Experimental

2.1. Materials and methods

All chemicals and solvents were in analytical grade and used as commercially available. 3-Carboxylsalicylidene was prepared by the literature method [27]. Elemental analyses for C, H and N were carried out on a Perkin-Elmer 2400II analyzer. The infrared spectra were recorded on an Avater-360 spectrometer using KBr pellets in a range of 400–4000 cm⁻¹. Thermogravimetric analysis was carried out on a TGA/SDTA851^e analyzer in a nitrogen atmosphere, and the complexes were heated to 1000 °C at a heating rate of 10 °C min⁻¹. Magnetic measurement was carried out on polycrystalline samples with a MPMS-7SQUID magnetometer. Diamagnetic corrections were made with Pascal's constants for all atoms.

2.2. Synthesis of the Schiff base ligand (H_4L^1)

A solution of *N*-(2-aminoethyl)-1,2-ethanediamine (0.515 g, 5 mmol) and 3-carboxylsalicylidene (1.66 g, 10 mmol) in methanol (20 mL) was heated at 70 °C for 2 h. After the solution was cooled to room temperature, the yellow precipitates were collected by filtration. Yield: 0.88 g (70%). *Anal.* Calc. for $C_{20}H_{21}N_3O_6$: C, 60.15; H, 5.26; N, 10.53. Found: C, 60.04; H, 5.41; N, 10.46%. IR (KBr, cm⁻¹): $\nu_{C=N}$ 1602 cm⁻¹, ν_{N-H} 3436 cm⁻¹, ν_{COOH} 1651 cm⁻¹.

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Scheme 1. Proposed mechanism for the formation of H_2L^2 .

2.3. Synthesis of $\{[CuMnL^1][CuL^2]\}_2 \cdot 2CH_3OH(1)$

 H_4L^1 (0.398 g, 1 mmol) and NaOH (0.16 g, 4 mmol) were dissolved in 20 mL methanol with stirring, then $Cu(OAC)_2 \cdot 6H_2O$ (0.18 g, 1 mmol) and $Mn(CIO_4)_2 \cdot 6H_2O$ (0.37 g, 1 mmol) was added. After heating to reflux for two hours, the resultant dark brown solution was filtered, the filtrate was left to stand at room temperature. Green crystals of **1** were obtained in one month. Yield: 0.71 g (40%). *Anal.* Calc. for $C_{66}H_{76}Cu_4Mn_2N_{12}O_{22}$: C, 45.18; H, 4.33; N, 9.58. Found: C, 45.23; H, 4.19; N, 9.49%. IR (KBr, cm⁻¹): $v_{C=N}$ 1601 cm⁻¹, v_{N-H} 3397 cm⁻¹, v_{COO} - 1644 cm⁻¹.

2.4. X-ray crystallography

The single crystal used for data collection was selected and mounted on a Bruker Smart APEX diffractometer with a CCD detector using graphite monochromated Mo K α radiation ($\lambda = 0.071073$ nm). Lorentz and polarization factors were made for the intensity data and absorption corrections were performed using sADABS [28]. The crystal structures were solved using SHELXTL and refined using full matrix least-squares [29]. The hydrogen atom positions were calculated theoretically and included in the final cycles of refinement in a riding model along with attached carbon atoms. Crystal data collection and refinement parameters are given in Table 1. The selected bond lengths and angles are given in Table 2.

3. Results and discussion

3.1. Crystal structure of complex 1

Single-crystal X-ray diffraction analysis reveals that complex 1 is a novel heterohexanuclear structure. The asymmetry unit of complex **1** contains a trinuclear [CuMnL¹][CuL²]moieties (Fig. 1). The result reveals that one imine bond in the ligand H₄L¹ is degraded into NH₂ group, giving the new ligand H₂L². According to the idea proposed earlier by Parr and co-workers [30], coordination and so deprotonation of one arm of the ligand H_4L^1 to Cu^{2+} possibly forms small quantities of $H_3O^+ClO_4^-$ in situ, which catalyses the hydrolysis of the remaining unbound 3-carboxylsalicylidene arms back to amine groups, then bind to the metal center. In the [CuL²] unit, the Cu1 atom has five coordinated square pyramidal, CuN₃O₂, surrounding. Three nitrogen atoms (N1,N2,N3) and a oxygen atom (O3) from the ligand L^2 build the basal plane, whereas the apical position is occupied by a oxygen atom (O9) from another ligand L¹. The Cu–O bond lengths range from 1.964(3) to 2.407(4) Å, and Cu–N bond lengths range from 1.952(4) to 2.011(4) Å. In the [CuMnL¹] unit, the coordination polyhedron of

able 1
Crystal data and structure refinement for complexes 1.

Complex	1
Empirical formula	C ₆₆ H ₇₆ Cu ₄ Mn ₂ N ₁₂ O ₂₂
Formula weight	1753.43
T (K)	298(2)
Wavelength (nm)	0.071073
Crystal system	triclinic
Space group	ΡĪ
Unit cell dimensions	
a (Å)	10.0958(6)
b (Å)	13.3245(8)
c (Å)	13.5004(8)
α (°)	96.922(1)
β(°)	106.486(1)
γ (°)	90.396(1)
V (Å ³)	1727.1(2)
Ζ	1
$D_{\text{calc}} (\text{g cm}^{-3})$	1.686
$\mu (\mathrm{mm}^{-1})$	1.651
F(000)	898
Crystal size/mm ³	$0.26\times0.18\times0.15$
θ range (°)	1.54-25.10
Independent reflections	$6083 \ (R_{\rm int} = 0.0122)$
Goodness-of-fit (GOF) on F ²	1.073
Final R indices $[I > 2\sigma(I)]$	$R_1 = 0.0393$, $wR_2 = 0.1003$
R indices (all data)	$R_1 = 0.0454, wR_2 = 0.1039$

 ${R_1} = \sum ||F_0| - |F_c|| / \sum |F_0|, \ wR_2 = [\sum w(|F_0|^2 - |F_c|^2)^2 / \sum w(F_0^2)^2]^{1/2}.$

Cu2 also can be considered as a square pyramid with apical elongation (4 + 1). The basal plane of Cu2 is formed by two nitrogen atoms (N5, N6) and two oxygen atoms (O6, O7) from the ligand L¹. The axial position is occupied by a water molecular. The Cu-N distances are distributed in the range 1.928(4)-1.983(4) Å, and the Cu–O distances are in the range 1.906(3)–1.977(3) Å. The axial (Cu2-O1W) bond length is 2.414(5) Å, which is elongated due to the Jahn–Teller effect of the d⁹ electronic configuration. The Mn1 ion exhibits a distorted octahedral geometry. The equatorial plane is described by there oxygen atoms (O5, O6, O8) from the ligand L¹ and a oxygen atom (O2) from the ligand L². The Mn-O bond lengths range from 2.089(3) to 2.177(3) Å. The axial positions are filled with two oxygen atoms (O3, O7) with elongated Mn-O distances (Mn1–O3 = 2.258(3), Mn1–O7 = 2.246(3) Å). The Cu1···Mn1 and Cu2···Mn1 distances are 3.370 and 3.177 Å, respectively. The Cu–O–Mn angle vary from 99.53° to 105.70°.

From Fig. 2, we can find that the adjacent trinuclear units are bridged together by a oxygen atom (O9) of ligand L^1 coordinates to Cu1 ions to form a novel heterohexanuclear Cu₄^{II}Mn₂^{II} structure.

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

	.,	-	
Cu1-N1	1.952(4)	Cu1-N2	2.006(4)
Cu1-03	1.964(3)	Cu1-N3	2.011(4)
Cu1-09#1	2.407(4)	Cu2-07	1.906(3)
Cu2-N6	1.928(4)	Cu2-N5	1.983(4)
Cu2-06	1.977(3)	Cu2-01W	2.414(5)
Mn1-08	2.089(3)	Mn1-05	2.090(3)
Mn1-02	2.104(4)	Mn1-06	2.177(3)
Mn1-07	2.246(3)	Mn1-03	2.258(3)
N1-Cu1-O3	92.4 (2)	N1-Cu1-N2	83.3(2)
03-Cu1-N2	161.9(2)	N1-Cu1-N3	166.1(2)
03-Cu1-N3	101.3(2)	N2-Cu1-N3	84.4(2)
04-Cu1-03	84.6(3)	04-Cu1-N4	169.5(3)
N1-Cu1-O9#1	83.8(2)	03-Cu1-09#1	104.0(2)
N2-Cu1-O9#1	93.0(2)	N3-Cu1-09#1	90.6(2)
07-Cu2-N6	93.9(2)	07-Cu2-06	85.4(1)
N6-Cu2-O6	157.6(3)	07-Cu2-N5	176.4(2)
N6-Cu2-N5	82.6(2)	06-Cu2-N5	98.3(2)
07-Cu2-01W	85.5(2)	N6-Cu2-01W	109.8(2)
06-Cu2-01W	92.4(2)	N5-Cu2-01W	94.6(2)
08-Mn1-05	97.4(1)	08-Mn1-02	93.3(2)
05-Mn1-02	159.6(1)	08-Mn1-06	150.2(1)
05-Mn1-06	83.1(1)	02-Mn1-06	96.1(1)
08-Mn1-07	78.8(1)	05-Mn1-07	109.6(1)
02-Mn1-07	89.4(1)	06-Mn1-07	73.1(1)
08-Mn1-03	96.6(1)	05-Mn1-03	82.8(1)
02-Mn1-03	78.8(1)	06-Mn1-03	112.9(1)
07-Mn1-03	167.2(1)	Cu1-03-Mn1	105.70(14)
Cu2-06-Mn1	99.69(13)	Cu2-07-Mn1	99.53(13)

Symmetry codes: #1 - x + 2, -y, -z + 1.



Fig. 1. The asymmetric unit of the complex 1. Hydrogen atoms are omitted for clarity.



Fig. 2. The heterohexanuclear $Cu_4^{II}Mn_2^{II}$ structure of complex **1**. The methanol molecules and hydrogen atoms are omitted for clarity. Symmetry codes: #1 –*x* + 2, –*y*, –*z* + 1.

3.2. Thermogravimetric analysis

The thermal property of **1** has been measured in the N₂ flowing atmosphere with the heating rate of 10 °C min⁻¹ from 25 to 1000 °C. The TGA curve of **1** can be divided into three steps of weight loss. The weight loss of 5.61% during the first step from 25 to 202 °C corresponds to the release of two methanol molecules and two coordination water molecules (Calc. 5.70%). The following weight loss of 22.83% occurs from 288 to 325 °C which is attributed to the loss of the ligand L² (Calc. 23.00%). Finally, the decomposition of complex 1 began at 455 °C.

3.3. Magnetic properties of complex 1

The magnetic behavior of complex **1** is investigated in the 2– 300 K temperature range and indicative of an overall anti-ferromagnetic coupling, as seen in Fig. 3. At room temperature, the $\chi_M T$ value is 10.03 cm³ mol⁻¹ K, which is lower than the spin-only value (10.25 cm³ mol⁻¹ K) for four uncoupled copper(II) (*S* = 1/2) and two high spin manganese(II) (*S* = 5/2) spin systems. Upon cooling the $\chi_M T$ values decrease regularly, approaching a minimum around 2 K with $\chi_M T$ = 3.48 cm³ mol⁻¹ K.

In the hexanuclear complex system, there are three kinds of magnetic interaction, namely: (i) Cu1–Mn1 through phenoxo-oxygen and carboxyl oxygen bridges; (ii) Cu2–Mn1 through phenoxo-oxygen bridges; (iii) Cu1#1–Mn1 through carboxyl oxygen bridge. Taking into account all the three interaction in the Hamiltonian given in Eq. (1) and using the computational program of MAGPACK [31,32], we have obtained an excellent simulation of the experimental data from the following parameters $J_1 = -11.2$ cm⁻¹, $J_2 = -3.9$ cm⁻¹, $J_3 = 0.09$ cm⁻¹ and g = 2.04 (Fig. 3).

$$\hat{H} = -2J_1 \left(\widehat{S}_{Cu2} \widehat{S}_{Mn1} + \widehat{S}_{Cu2A} \widehat{S}_{Mn1A} \right) - 2J_2 \left(\widehat{S}_{Cu1} \widehat{S}_{Mn1} + \widehat{S}_{Cu1A} \widehat{S}_{Mn1A} \right) - 2J_3 \left(\widehat{S}_{Cu1} \widehat{S}_{Mn1A} + \widehat{S}_{Cu1A} \widehat{S}_{Mn1} \right)$$
(1)

In order to understand the magnetic exchange mechanism, it is important to note that complex **1** has two monatomic bridges (from carboxylate and phenoxide) and a *syn–anti* carboxylate bridge. For the carboxylate bridge, it is well known that its presence in a *syn–anti* conformation causes weak ferromagnetic or antiferromagnetic interactions [33–37]. The nature of magnetic interaction is very dependent on the bond angle M–O–M for the monatomic bridge. This dependence has been well-studied for Cu^{II} polynuclear complexes. For other metal ions or heteropolynuclear complexes, this dependence is quite unknown. In complex **1**, the Cu–O–Mn angle vary from 99.53° to 105.70°, which is similar to



Fig. 3. χ_M vs. *T* and $\chi_M T$ vs. *T* plots for complex **1**.

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the value reported in the literature [38-42]. As expected, the interactions between metal ions in the trinuclear moiety are all antiferromagnetic.

4. Conclusion

In summary, a novel heterohexanuclear Cu₄^{II}Mn₂^{II} complex has been synthesized and characterized structurally. Furthermore, magnetic properties of **1** have been also measured and analyzed. This work may provide a helpful strategy for the construction of other new heteropolynuclear with fancy structures and amusing properties, which is undergoing in our laboratory.

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Appendix A. Supplementary material

CCDC 824122 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/ data_request/cif. Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/ j.ica.2012.03.049.

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