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## Synthesis, crystal structure, luminescence and electrochemical properties of a Salamo-type trinuclear cobalt(II) complex

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**Abstract:** A trinuclear Co(II) complex, [{CoL(C,H,OH)},- $(OAc)_{CO} \cdot C_{H}NO$ , was synthesized by the reaction of a Salamo-type chelating ligand (H,L=4,42'-dinitro-2,2'-[1,2-ethylenedioxybis(nitrilomethylidyne)]diphenol) with cobalt(II) acetate tetrahydrate in *n*-butanol, and characterized by elemental analyses, X-ray crystallography, FT-IR and UV/Vis spectra. In the Co(II) complex, there are two ligand L<sup>2–</sup> units, two  $\mu_2$ -acetate ions, two coordinated *n*-butanol molecules and one non-coordinated N,N-dimethylformamide molecule. The Co(II) atoms in the structure of the Co(II) complex adopt slightly distorted octahedra geometries. Furthermore, through intermolecular C–H · · · O, O–H · · · O and C–H · · ·  $\pi$  interactions, infinite layer-like, plane-like and 3D supramolecular structures are constructed. The fluorescence and electrochemical properties of the Co(II) complex have also been investigated.

**Keywords:** complex; crystal structure; property; Salamotype ligand; synthesis.

## **1** Introduction

Many chemists have focused their studies mainly on the investigation of the syntheses and crystal structures of metal complexes having the Salen ligand and its analogues [1–7]. These complexes can be used in lumines-cence materials [8–13], biochemistry [14–21], magnetic devices [22–25], optical sensors [26–30], catalysis [20, 31], and supramolecular architectures [32–41]. Electrochemical properties are also important in modern coordination

Chemical and Biological Engineering, Lanzhou Jiaotong University, Lanzhou, Gansu 730070, P. R. China chemistry aiming at potential applications of Salentype compounds and their transition metal complexes [42–47].

Compared with Salen-type ligands, synthesis of Salamo-type ligands is more difficult. With an O-alkyloxime unit  $(-CH=N-O-(CH)_{2}-O-N=CH-)$  instead of the  $(-CH = N - (CH)_{2} - N = CH -)$  group, the high electronegativity of the oxygen atoms is expected to affect strongly the electronic properties of the N<sub>2</sub>O<sub>2</sub> coordination sphere, which can lead to the novel properties and structures of the resulting complexes. Furthermore, this variety can give better ways to control and monitor coordination in the context of the infinite supramolecular structures [48–57]. In order to study the structural features, the spectral characteristics and electrochemical properties of transition metal complexes with Salamo-type bisoxime ligands, we herein report one trinuclear Co(II) complex with 4,4'-dinitro-2,2'-[1,2-ethylenedioxybis(nitrilomethyli dyne)]diphenol (H<sub>2</sub>L).

## 2 Experimental section

#### 2.1 Materials and physical measurements

All chemicals were of analytical reagent grades and were used without further purification. C, H and N analyses were obtained using a VarioEL V3.00 automatic elemental analysis instrument. Elemental analyses for Co(II) ions were performed by an IRIS ER/S-WP-1 ICP atomic emission spectrometer. IR spectra were recorded on a VERTEX70 FT-IR spectrophotometer, with samples prepared as KBr (500–4000 cm<sup>-1</sup>) pellets. Melting points were obtained by the use of an X4 microscopic melting point apparatus produced by Beijing Taike Instrument Limited Company and were uncorrected. Fluorescent spectra were taken on an LS-55 fluorescence photometer. X-ray single crystal structure determinations were carried out on a Bruker Smart Apex CCD diffractometer. Cyclic voltammetry measurements were performed using a Chi 660 voltammetric analyzer in DMF containing 0.05 mol L<sup>-1</sup> tetrabutylammonium perchlorate.

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Scheme 1: Synthetic route to the Salamo-type ligand H<sub>2</sub>L.

## 2.2 Synthesis and characterization of the ligand H<sub>2</sub>L

The preparation step involved in the synthesis of  $H_2L$  is given in Scheme 1. 4,4'-Dinitro-2,2'-[1,2-ethylenedioxybis (nitrilomethylidyne)]diphenol ( $H_2L$ ) was prepared by the modification of the reported method [58–60]. A mixture of 5-nitrosalicylicaldehyde (341.6 mg, 2.01 mmol) and 1,2-bis(aminoxy)ethane (93.4 mg, 1.00 mmol) in ethanolic solution was stirred at 55°C for 5 h. After cooling to room temperature, the precipitate was filtered, and washed successively with ethanol and ethanol/hexane (1:4). The product was dried under vacuum to give 298 mg of colorless microcrystals. Yield: 85%. m.p. 202–203°C. – Elemental analysis for  $C_{16}H_{14}N_4O_8$ : calcd. C 49.24, H 3.62, N 14.35; found C 49.12, H 3.75, N 14.12.

#### 2.3 Synthesis of the Co(II) complex

A solution of  $Co(OAc)_2$ ·4H<sub>2</sub>O (2.47 mg, 0.01 mmol) in *n*-butanol (3 mL) was added to a solution of H<sub>2</sub>L (3.52 mg, 0.01 mmol) in chloroform (3 mL) at room temperature. The resulting solution immediately turned yellow, and was stirred for 2 h at ambient temperature. The solution was filtered and the filtrate was allowed to stand at room temperature for about 4 weeks. The solvent was partially evaporated to obtain several red prismatic single crystals suitable for X-ray crystallographic analysis. – Elemental analysis for C<sub>50</sub>H<sub>64</sub>Co<sub>3</sub>N<sub>10</sub>O<sub>24</sub>: calcd. C 43.97, H 4.72, N 10.25, Co 12.94; found C 43.76, H 4.89, N 10.09, Co 12.71.

#### 2.4 X-ray structure determination of the Co(II) complex

A suitable single crystal of the Co(II) complex with approximate dimensions of  $0.24 \times 0.22 \times 0.02$  mm<sup>3</sup> was placed on a Bruker Smart 1000 CCD area detector. The diffraction data were collected using a graphite monochromated  $MoK_{\alpha}$  radiation ( $\lambda = 0.071073$  nm) at 291(2) K. The structure was solved the using the program SHELXS-97 and difference Fourier techniques, and refined by full-matrix leastsquares methods on  $F^2$  using SHELXL-97 [61]. Hydrogen atoms were added in idealized positions. Details of the data collection and the refinement are collected in Table 1.

CCDC 1567223 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.

### **3** Results and discussion

#### 3.1 IR spectra of H<sub>2</sub>L and its Co(II) complex

The FT-IR spectra of  $H_2L$  and its corresponding Co(II) complex show various bands in the range of

 Table 1: Crystal data and structure refinements for the Co(II) complex.

Co(II) complex	[{CoL(C <sub>4</sub> H <sub>9</sub> OH)} <sub>2</sub> (OAc) <sub>2</sub> Co] ·		
	C <sub>3</sub> H <sub>7</sub> NO		
Formula	C <sub>50</sub> H <sub>64</sub> Co <sub>3</sub> N <sub>10</sub> O <sub>24</sub>		
Formula weight, g∙mol⁻¹	1365.90		
Temperature, K	291(2)		
Wavelength, Å	0.71073		
Crystal system	Monoclinic		
Space group	P2,/c		
Unit cell dimensions	-		
<i>a</i> , Å	10.850(4)		
<i>b</i> , Å	15.993(2)		
<i>c</i> , Å	17.724(2)		
β, °	102.41(3)		
Volume, ų	3003.7(13)		
Ζ	2		
$D_{c}$ , g cm <sup>-3</sup>	1.51		
$\mu$ , mm <sup>-1</sup>	0.9		
F(000), e	1414		
Crystal size, mm <sup>3</sup>	$0.24 \times 0.22 \times 0.02$		
$ heta$ range, $^\circ$	1.922-25.999		
hkl range	-10, 13/-18, 19/-21, 12		
Reflections collected	12 248		
Independent reflections/R <sub>int</sub>	5908/0.0239		
Completeness to $\theta_{\max}$ , %	99.8		
Data/restraints/parameters	5908/1/398		
Final <i>R</i> 1/ <i>wR</i> 2 [ <i>l</i> > 2 <i>σ</i> ( <i>l</i> )] [ <i>l</i> > 2 <i>σ</i> ( <i>l</i> )]	0.0475/0.1291		
Final R1/wR2 (all data)	0.0655/0.1358		
Goodness-of-fit (F <sup>2</sup> )	1.010		
$\Delta  ho_{ m max/min}$ , $e$ Å <sup>-3</sup>	0.820/-0.484		

400–4000 cm<sup>-1</sup> so as to identify frequencies owing to the Co–O and Co–N bonds (Fig. 1). The free ligand H<sub>2</sub>L shows a characteristic C = N stretching band at 1627 cm<sup>-1</sup>, while the C = N stretching band of the Co(II) complex is observed at 1606 cm<sup>-1</sup>. The shift to lower frequency by ca. 21 cm<sup>-1</sup> upon complexation shows a decrease in the C=N bond order due to the coordinative bonds of the Co(II) atom with the oxime nitrogen lone pair [28]. The Ar–O stretching band occurs at 1274 cm<sup>-1</sup> for H<sub>2</sub>L, but is observed at 1242 cm<sup>-1</sup> for the Co(II) complex; shift to lower frequency indicates that Co–O bonds are formed between the Co(II) atoms and the phenolic oxygen atoms [28]. Besides, an O–H stretching band appears at 3444 cm<sup>-1</sup> in the Co(II) complex, which is evidence of the existence of the coordinated *n*-butanol molecules [36].

# **3.2 UV/Vis absorption spectra of H**<sub>2</sub>L and its Co(II) complex

UV/Vis absorption spectra of H<sub>2</sub>L and its corresponding Co(II) complex in CH<sub>3</sub>OH solutions at 298 K are shown in Fig. 2. The spectrum of H<sub>2</sub>L shows three absorption peaks at ca. 221, 267 and 322 nm [37–39]. The former two can be assigned to the  $\pi$ – $\pi$ \* transition of the benzene rings, and the latter one to intra-ligand  $\pi$ – $\pi$ \* transitions of the C=N bonds [40–42]. Upon the addition of Co(II) ions, the peaks at 267 and 322 nm disappear. Furthermore, compared with the absorption peaks of the free ligand, the corresponding peaks of the Co(II) complex are observed at ca. 227 nm. The bathochromic shift indicates the coordination of the Co(II) ions with the ligand. The UV/Vis titration showed



**Fig. 1:** IR spectra of H<sub>2</sub>L and its corresponding Co(II) complex.



Fig. 2: UV/Vis absorption spectra of H<sub>2</sub>L and its Co(II) complex.

that the stoichiometry of complexation between them is 3:2. The appearances of four distinct isosbestic points at 330, 312, 280 and 261 nm are a significant evidence of a complete deprotonation of the phenolic hydrogen present in the Co(II) complex and a successful complexation between the ligand and the Co(II) ions.

#### 3.3 Description of the crystal and molecular structure of the Co(II) complex

The molecular structure of the Co(II) complex is shown in Fig. 3. Selected bond lengths and angles are listed in Table 2.

The Co(II) complex crystallizes in the monoclinic, space group  $P2_1/c$  with Z=2. It consists of three Co(II) atoms, two deprotonated L<sup>2–</sup> units, two  $\mu_2$ -acetate ions, two coordinated n-butanol molecules and one noncoordinated N,N-dimethylformamide molecule resulting in a trinuclear Co(II) complex. As there are two of these complexes in the unit cell, the entire trinuclear aggregate has crystallographic inversion  $(\overline{1})$  symmetry. All the sixcoordinated Co(II) atoms lie in a slightly distorted octahedral coordination environment. The two terminal Co(II) (Co1 and Co1<sup>#1</sup>) atoms are located in the N<sub>2</sub>O<sub>2</sub> coordination sphere of L<sup>2-</sup>. One oxygen atom (O9 or O9<sup>#1</sup>) comes from the acetato bridge and the other oxygen atom (O11 or O11<sup>#1</sup>) comes from the *n*-butanol molecule. The coordination sphere of the central Co(II) atom (Co<sub>2</sub>) contains four phenol oxygen atoms (01, 06, 01<sup>#1</sup> and 06<sup>#1</sup>) from two L<sup>2-</sup> and two oxygen atoms (O10 and O10<sup>#1</sup>) from two bridging  $\mu_2$ -acetate anions. As do the ligands L, these acetate anions firmly link the peripheral Co(II) atoms with the central one.



Fig. 3: (a) Molecular structure of the Co(II) complex (displacement ellipsoids at 30% probability, the hydrogen atoms are omitted for clarity). (b) The coordination polyhedra for Co(II) atoms of the Co(II) complex.

#### 3.4 Supramolecular interaction of the Co(II) complex

Notably, supramolecular interactions exist in crystals of the Co(II) complex. The hydrogen bond parameters are summarized in Table 3. As is shown in Fig. 4, there are three pairs of intramolecular hydrogen bonding interactions (C3-H3····O2, C8-H8A····O9 and C15-H15····O10) [62-64]. Besides, the structure is stabilized by intermolecular C–H···O, O–H···O and C–H··· $\pi$  hydrogen bond interaction. As a result, a 3D supramolecular structure is formed (Fig. 5; Figs. S1 and S2 in the Supplementary Information). The 3D supramolecular structure of the Co(II) complex is composed of two parts. The first part is linked by intermolecular  $C-H\cdots O$  and  $O-H\cdots O$  hydrogen bonding interactions. The other part is composed of the C–H  $\cdots \pi$ interactions. The intermolecular hydrogen bonds of the Co(II) complex not only make its structure variable but also make it stable [65-68].

#### 3.5 Fluorescence properties of the Co(II) complex

The emission spectra of the Co(II) complex in dilute DMF solution at ambient temperature are shown in Fig. 6. The H<sub>L</sub> ligand exhibits an intense photoluminescence with maximum emission at ca. 435 nm when excited at 320 nm,

Bond		Bond		Bond	
Co1-01	2.042(2)	Co2-01	2.161(2)	Co1-N2	2.120(2)
Co1-06	2.065(2)	Co2-06	2.109(2)	Co1-N3	2.110(3)
Co1-09	2.065(2)	Co2-010	2.052(2)	Co2-06#1	2.1089(19)
Co1-011	2.107(2)	Co2-01#1	2.161(2)	Co2-010#1	2.052(2)
Angle		Angle		Angle	
01-Co1-O6	81.45(9)	011-Co1-N2	85.74(10)	06-Co1-09	93.94(9)
01 <sup>#1</sup> -Co2-O10 <sup>#1</sup>	87.62(9)	01#1-Co2-O6#1	77.73(8)	06-Co1-011	89.35(9)
01-Co1-09	91.89(9)	011-Co1-N3	87.41(10)	06-Co1-N2	167.41(9)
06 <sup>#1</sup> -Co2-O10 <sup>#1</sup>	87.23(8)	N2-Co1-N3	104.98(12)	01#1-Co2-O6	102.27(8)
01-Co1-011	89.13(9)	01-Co2-O6	77.73(8)	06-Co1-N3	86.34(11)
01-Co1-N2	86.88(10)	01-Co2-010	87.62(9)	01-Co2-010#1	92.38(9)
09-Co1-N3	92.26(10)	01-Co2-O6#1	102.27(8)	01#1-Co2-010	92.38(9)
09-Co1-N2	91.14(10)	09-Co1-011	176.66(9)	06-Co2-010	87.23(8)
01-Co1-N3	167.35(12)	06 <sup>#1</sup> -Co2-O10	92.77(8)	06-Co2-010 <sup>#1</sup>	92.77(8)

Table 2: Selected bond lengths (Å) and angles (deg) for the Co(II) complex.<sup>a</sup>

<sup>a</sup>Symmetry transformation used to generate equivalent atoms: #1-x, 1-y, 1-z.

Table 3: Hydrogen bonding interactions (Å, deg) for the Co(II) complex.<sup>a</sup>

D–H···A	d(D–H)	d(H···A)	d(D···A)	∠DHA	Symmetry code A
С3-Н3···02	0.93	2.37	2.687(4)	100	х, <i>y</i> , z
C8-H8A···09	0.97	2.45	3.292(4)	145	x, y, z
C15-H15…010	0.93	2.56	3.228(4)	129	-x, 1-y, 1-z
011-H1102	0.93	2.60	3.462(4)	155	1-x, $1/2+y$ , $3/2-z$
011-H11…03	0.93	2.16	3.003(4)	150	1-x, $1/2+y$ , $3/2-z$
С9-Н9А08	0.97	2.36	3.087(5)	131	1+ <i>x</i> , <i>y</i> , <i>z</i>
С9-Н9В…02	0.97	2.39	3.300(5)	156	1-x, $1/2+y$ , $3/2-z$
C17-H17A···O12	0.97	2.49	3.287(5)	139	1-x, $1/2+y$ , $3/2-z$
C18-H18A···O3	0.97	2.49	3.206(5)	130	1-x, $1/2+y$ , $3/2-z$
C22-H22C…07	0.96	2.54	3.484(5)	167	x, 3/2 - y, -1/2 + z
C24-H24A…05	0.96	2.28	3.109(6)	144	x, -1 + y, z
C24-H24B···O3	0.96	2.59	3.438(6)	147	1-x, $-1/2+y$ , $3/2-z$
C25-H25···O2	0.93	2.48	3.027(5)	118	x, y, z
C8–H8B…Cg1		2.89		123	1 - x, 1 - y, 1 - z
C18-H18BCg2		2.80		154	<i>x</i> , <i>y</i> , <i>z</i>

<sup>a</sup>Cg1 and Cg2 are the centroids of the C1–C6 and C11–C16 atoms, respectively.

which can be assigned to intraligand  $\pi - \pi^*$  transitions. Compared with H<sub>2</sub>L, weaker fluorescence intensity of the Co(II) complex is observed, indicating that the fluorescence characteristic has been influenced by the introduction of the Co(II) ions [41, 50].

## 3.6 Electrochemistry studies of the Co(II) complex

The cyclic voltammogram of the Co(II) complex is shown in Fig. 7. The cyclic voltammetry was performed within the potential range -1.5 to +1.5 V at the scanning rate of 50 mV s<sup>-1</sup> at ambient temperature (300 K) in a standard three-electrode cell, consisting of a glassy carbon (GC) disk (U=5 mm) as a working electrode, a platinum wire as auxiliary and a Ag/AgCl electrode as reference. In DMF, the Co(II) complex displays two pairs of redox peaks resulting from the redox reactionds of Co<sup>III</sup>/Co<sup>II</sup> and Co<sup>II</sup>/Co<sup>I</sup>. The first pair have potentials *E*pa<sub>1</sub> of -0.762 V and *E*pc<sub>1</sub> of -0.870 V for the currents  $ipa_1=49.101$  µA and  $ipc_1=-89.764$  µA. Another pair of redox peaks appear for the electron transfer between Co<sup>II</sup> and Co<sup>I</sup> with potentials *E*pa<sub>2</sub> of -1.754 V and *E*pc<sub>2</sub> of -1.269 V for currents  $ipa_2=-9.520$  µA,  $ipc_1=-88.079$  µA. Also, these differences can be associated with the coordination environment around the metal center. These characteristics suggest that the electrolysis processes of the Co(II) complex are irreversible.



Fig. 4: Intramolecular hydrogen bonding interactions of the Co(II) complex.



**Fig. 5:** Part of the infinite layer-like of the Co(II) complex with intermolecular hydrogen bondings (hydrogen atoms, except those forming hydrogen bonds, are omitted for clarity).



Fig. 6: Fluorescence spectra of the Co(II) complex.



Fig. 7: Cyclic voltammogram of the Co(II) complex in DMF solution at 293 K. Scan rate: 50 mV s<sup>-1</sup>.

### **4** Conclusions

We have designed and synthesized a supramolecular trinuclear Co(II) complex with a Salamo-type ligand H<sub>2</sub>L. The Co(II) complex has been characterized by physicochemical methods and single-crystal X-ray diffraction. In the Co(II) complex, the three Co(II) atoms are hexa-coordinated by the O and N atoms of two completely deprotonated L<sup>2-</sup> units, two  $\mu_2$ -acetato anions and two coordinated n-butanol molecules. In addition, supramolecular structures ranging from chains to 3D arrays are formed by abundant noncovalent interactions. The luminescent properties of H<sub>2</sub>L and its Co(II) complex were also investigated, compared with H<sub>2</sub>L, the Co(II) complex exhibits lower fluorescence intensity. Electrochemistry studies suggest that electrolysis processes are irreversible.

## **5** Supporting information

Figs. S1 and S2 of the supramolecular structure are available online (https://doi.org/10.1515/znb-2017-0191).

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