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A dinuclear nickel(II) complex derived from an asymmetric Salamo-type N_2O_2 chelate ligand: synthesis, structure and optical properties

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Abstract: A new dinuclear Ni(II) complex, [{Ni(L) (C_5H_5N) }], has been synthesized with 4-chloro-4',6'-dibromo-2,2'-[ethylenedioxybis(nitrilomethylidyne)] diphenol (H₂L), and characterized by elemental analyses, FT-IR, UV/Vis, molar conductance and X-ray crystallographic analysis. Each Ni(II) atom is located at a N₂O₂ coordination site of a completely deprotonated (L)²⁻ unit. Two μ -phenoxo oxygen atoms from two [Ni(L)] units and two pyridine nitrogen atoms coordinate with two Ni(II) atoms. The two hexa-coordinated Ni(II) atoms have slightly distorted octahedral geometries. The μ -phenoxo bridges play important roles in assembling Ni²⁺ and (L)²⁻ units. This 2:2 (Ni²⁺:(L)²⁻) structure is different from that of other Salamotype Ni(II) complexes reported earlier.

Keywords: asymmetric Salamo-type ligand; crystal structure; Ni(II) complex; optical properties; synthesis.

1 Introduction

Transition metal complexes bearing salen-type ligands $(R-CH=N-(CH)_2-N=CH-R)$ [1–4] have been extensively investigated as catalysts [5–8], models of reaction centers of metalloenzymes [9, 10], nonlinear optical materials [11, 12], magnetic materials [13–16] and biological models for understanding the structures of biomolecules and biological processes [17–19]. Although some progress has been made in the studies of salen–Ni(II) complexes, it still seems that there could be new and specific applications for such a unique group of compounds. To change the structures or improve the functions of the resulting complexes, chemical modifications of the ligand, e.g. introduction of some

functional groups or substitution of some parts, should be effective. In particular, replacement of some atoms of the ligand with other elements often drastically changes its properties [20-23]. Where an O-alkyl oxime moiety $(-CH=N-O-(CH_2)_n-O-N=CH-)$ is used instead of the imine moiety, the larger electronegativity of the oxygen atoms is expected to affect strongly the electronic properties of the N_2O_2 coordination sphere, and to lead to new properties and structures of the resulting complexes [24–29]. The structures of Salamo-type Ni(II) complexes reported are mono-[30, 31], tri- [26, 31, 32] and hepta-nuclear [33]. Surprisingly, to the best of our knowledge, no other structures of multinuclear Ni(II) complexes have yet been reported.

Herein, following our previous studies on transition metal Salamo-type complexes [34], we report the synthesis, characterization, supramolecular structure and optical properties of a new dinuclear Ni(II) complex with an asymmetric Salamo-type N_2O_2 bisoxime chelate ligand (H₂L).

2 Experimental section

2.1 Materials

3,5-Dibromo-2-hydroxybenzaldehyde (>99%) and 2-hydroxy-5-chlorobenzaldehyde (>99%) were purchased from Alfa Aesar and used without further purification. 1,2-Bis(aminooxy)ethane was synthesized according to an analogous method reported earlier [20]. The other reagents and solvents were analytical grade reagents from Beijing Chemical Reagent Factory, and were used without further purification.

2.2 Methods

Elemental analysis for Ni was carried out with an IRIS ER/S·WP-1 ICP atomic emission spectrometer, and C, H and N analyses were carried out with a GmbH VarioEL V3.00 automatic elemental analyzer. FT-IR spectra were recorded on a VERTEX70 FT-IR spectrophotometer, with samples prepared as KBr (500–4000 cm⁻¹) and CsI

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Scheme 1: The synthetic route to H₂L.

(100–500 cm⁻¹) pellets. UV/Vis absorption spectra were recorded on a Shimadzu UV-2550 spectrometer. Molar conductance measurement was carried out on a model DDS-11D-type conductivity bridge using 1.0×10^{-3} mol L⁻³ solution in DMF at 25°C. Thermogravimetry-differential thermal analysis (TG-DTA) were carried out at a heating rate of 5°C min⁻¹ on a ZRY-1P thermoanalyzer. X-ray single crystal structure analysis was carried out on a Rapid Auto Version 3.0 Rigaku RAXIS-RAPID instrument. Melting points were measured by the use of a microscopic melting point apparatus from Beijing Taike Instrument Limited Company, and are uncorrected.

2.3 Synthesis and characterization of the ligand H₂L

The synthetic route to the ligand H₂L is shown in Scheme 1.

 $\rm H_2L$ was synthesized according to an analogous method reported earlier [26]. Yield: 81%. m.p. 128–129°C. – Elemental analysis for $\rm C_{16}H_{13}Br_2ClN_2O_4$ (492.55): calcd. C 39.02, H 2.66, N 5.69; found C 39.28, H 2.42, N 5.43.

2.4 Synthesis of the Ni(II) complex

A solution of Ni(OAc), ·4H, O (4.99 mg, 0.02 mmol) in ethanol (2 mL) was added dropwise to a solution of H₂L (9.87 mg, 0.02 mmol) in methanol (2 mL) at room temperature. The color of the mixture turned green immediately; 2 mL of acetone and two drops of pyridine were added. The solution was then stirred for 1 h at room temperature, placed in an oven and heated for 16 h at 60°C. The solution was kept still to clarify, and the supernatant was placed in an oven at 70°C for 3 days. The vessel was placed in an about 70°C water bath bottle stoppered with a cork and kept relatively sealed for about 1 week to obtain green block-like single crystals suitable for X-ray crystallographic analysis. Yield: 46%. The complex does not melt but decomposes at elevated temperatures. For details see below. - Elemental analysis for C42H32Br4Cl2Ni2N6O8 (1256.65): calcd. C 40.14, H 2.57, N 6.69, Ni 9.34; found C 40.11, H 2.58, N 6.70, Ni 9.36.

2.5 X-ray crystallography of the Ni(II) complex

A single crystal of the Ni(II) complex with approximate dimensions of $0.24 \times 0.32 \times 0.39$ mm³ was placed on a Bruker Smart diffractometer equipped with an Apex CCD area detector. The diffraction data were collected using graphite-monochromatized Mo K_{α} radiation ($\lambda = 0.71073$ Å) at 298(2) K. The structure was solved by using the program SHELXS-97 and difference Fourier techniques, and refined by full-matrix least squares on F^2 using SHELXL-97. All hydrogen atoms were added theoretically. The crystal and experimental data are shown in Table 1.

CCDC 1519393 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 Molar conductance of the Ni(II) complex

The Ni(II) complex is soluble in DMF and DMSO, but not soluble in ethanol, methanol, THF, acetone and acetonitrile. The free ligand is soluble in all of the aforementioned solvents. The molar conductance value of the Ni(II) complex at 25°C in 1.0×10^{-3} mol L⁻³ DMF solution is 5.7 Ω^{-1} cm² mol⁻¹. Comparing with the molar conductance in different types of electrolytes in organic solvents reported by W. J. Geary, we can conclude that the Ni(II) complex is a non-electrolyte [35].

3.2 IR spectra of H₂L and its Ni(II) complex

The free ligand H_2L exhibits Ar–O and C=N stretching bands at 1265 and 1610 cm⁻¹, which are shifted to lower frequencies by about 4 and 9 cm⁻¹ for the Ni(II) complex upon complexation. This effect results from the Ni–O and Ni–N interactions upon complexation similar to that

Table 1:	Crystal data and numbers pertinent to data collecti	on	and
structure	e refinement of the Ni(II) complex [{Ni(L)(C,H,N)}].		

Empirical formula	C ₄₂ H ₃₂ Br ₄ Cl ₂ Ni ₂ N ₆ O ₈
Formula weight	1256.62
Temperature, K	293(2)
Wavelength, Å	0.71073
Crystal size, mm ³	0.24×0.32×0.39
Crystal system	Orthorhombic
Space group	Pbca
Unit cell dimensions	
<i>a</i> , Å	14.5792(3)
<i>b</i> , Å	16.9377(4)
<i>c</i> , Å	18.0277(4)
Volume, Å ³	4451.73(17)
Ζ	4
$D_{\rm calcd}$, g cm ⁻³	1.8
μ , mm ⁻¹	6.9
<i>F</i> (000), e	2480.0
heta Range, deg	5.2-70.6
Index ranges <i>hkl</i>	$-13 \le h \le 17$
	$-16 \le k \le 20$
	− 15≤ <i>l</i> ≤21
Refl. collected/unique/R _{int}	10 931/4201/0.043
Completeness to $\theta = 25.00^{\circ}$, %	99.9
Data/restraints/ref. param.	4201/0/289
Final $R1/wR2$ [$I > 2\sigma(I)$]	0.0482/0.1331
Final <i>R</i> 1/ <i>wR</i> 2 (all data)	0.0426/0.0391
$\Delta\! ho_{ m max/min}$, e Å-3	0.83/-1.28



Fig. 1: UV/Vis absorption spectra of H_2L and its Ni(II) complex in DMF (5 × 10⁻⁵ mol L⁻¹).

shows ν (Ni–O) and ν (Ni–N) vibration absorption frequencies at 415 and 473 cm⁻¹, respectively. These assignments are consistent with the literature frequency values [33]. These new bands are not present in the spectrum of the free ligand H₂L. As pointed out by Percy and Thornton, the metal–oxygen and metal–nitrogen frequency assignments are at times very difficult [36, 37].

reported for other transition metal complexes [20, 26, 27, 32–34, 36].

The far-infrared spectrum of the Ni(II) complex was obtained in the region $500-100 \text{ cm}^{-1}$ in order to identify frequencies due to the Ni–O and Ni–N bonds. The spectrum

3.3 UV/Vis absorption spectra of H₂L and its Ni(II) complex

The UV/Vis absorption spectra of the ligand H_2L and its Ni(II) complex in diluted DMF solution are shown in Fig. 1.

Table 2: Selected bond lengths (Å) and angles (deg) for the title compound.^a

Bond		Bond		Bond	
Ni1-N1	2.063(3)	Ni1-N2	2.100(3)	Ni1-N3	2.115(3)
Ni1-01	2.004(2)	Ni1-04	2.051(2)	Ni1-04 ^{#1}	2.176(2)
Br1–C2	1.901(3)	Br2–C4	1.904(3)	Cl1-C13	1.754(4)
01-C1	1.275(4)	02-N1	1.439(4)	02-C8	1.428(5)
N2-C10	1.271(4)	N3-C17	1.338(4)	N3-C21	1.329(4)
C1-C2	1.429(5)	C1-C6	1.430(4)	C2-C3	1.369(5)
Angle		Angle		Angle	
01-Ni1-04	89.64(9)	01-Ni1-N1	87.44(10)	01-Ni1-N2	174.57(11)
01-Ni1-N3	88.45(10)	01-Ni1-04#1	88.85(8)	04-Ni1-N1	175.71(10)
04-Ni1-N2	85.73(10)	04-Ni1-N3	91.18(9)	04-Ni1-04#1	81.89(8)
N2-Ni1-N3	88.81(11)	N2-Ni1-N3#1	93.30(9)	N3-Ni1-04#1	172.58(9)
Ni1-01-C1	130.8(2)	Ni1-04-C16	122.89(18)	Ni1-04-Ni1 ^{#1}	98.11(8)
C16-04-Ni1#1	120.28(18)	Ni1-N1-02	121.54(19)	Ni1-N1-C7	127.7(2)
Ni1-N2-03	124.7(2)	Ni1-N2-C10	125.6(2)	Ni1-N3-C21	123.6(2)
Ni1-N3-C17	118.4(2)	01-C1-C2	120.8(3)	C2-C1-C6	114.1(3)

^aSymmetry transformations used to generate equivalent atoms: $^{#1}1-x$, 1-y, 1-z.



Fig. 2: (a) Molecular structure and atom numbering of the Ni(II) complex (hydrogen atoms are omitted for clarity). (b) Coordination polyhedra of the Ni(II) atoms.

The UV/Vis spectrum of the ligand H_2L exhibits two intense peaks at around 269 and 324 nm. The former absorption peak can be assigned to the π - π * transition of the benzene rings and the latter at 324 nm can be attributed to the intraligand π - π * transition of the oxime C=N group [38, 39].

The absorption peak of the free ligand at 269 nm shifts to the low energy region by about 5 nm in the Ni(II) complex, indicating the coordination of Ni²⁺ cations with (L)²⁻ anions. The absorption peak of the free ligand at 324 nm is absent in the Ni(II) complex. However, a new absorption peak is observed at 384 nm, which is assigned to that of an L \rightarrow M charge-transfer transition, which is characteristic for transition metal complexes in N₂O₂ coordination spheres [40].

3.4 Thermal properties

The thermal decomposition process of the Ni(II) complex can mainly be divided into two stages. First, the initial



Fig. 3: Intramolecular hydrogen bonding interactions of the Ni(II) complex (hydrogen atoms, except those forming hydrogen bonds, are omitted for clarity).

weight loss occurs in the range of 193.5-225.7°C, corresponding to an endothermic peak, and the TG curve shows that the weight loss corresponding to this temperature range is 12.3%, which roughly coincides with the value of 12.6%, calculated for the loss of two coordinated pyridine molecules. The Ni(II) complex has no melting point. The decomposing temperature of the Ni(II) complex (ca. 187°C) is higher than that of the ligand H₂L (ca. 153°C). It can be seen that the Ni(II) complex is more thermally stable than the uncomplexed ligand H₂L. Apparently its thermal stability is improved upon complexation to Ni(II). Next, the second weight loss starts at around 275.0°C. Subsequently, continuous mass loss was observed up to 800°C. At this temperature, NiO is formed. The total mass loss found (87.7%) was approximately consistent with that calculated (88.1%).

3.5 Structural description of the Ni(II) complex

X-ray crystallographic analysis has revealed the crystal structure of the Ni(II) complex. Selected bond lengths and

Table 3: Hydrogen bond parameters (Å, deg) for the Ni(II) complex.

D–H···A	d(D-H)	<i>d</i> (H···A)	<i>d</i> (D···A)	∠DHA	Symmetry code A
C9-H9B···O4	0.97	2.46	3.393(4)	160	1 - x, 1 - y,
					1 – <i>z</i>
C17-H17···O1	0.93	2.51	2.968(5)	110	
C20-H20···Cl1	0.93	3.15	3.817(4)	130	
C17-H17Br2	0.93	3.04	3.749(5)	134	



Fig. 4: View of the 2D supramolecular structure of the Ni(II) complex along the crystallographic *ac* plane (hydrogen atoms, except those forming hydrogen bonds, are omitted for clarity).

angles are given in Table 2. The molecular structure of the Ni(II) complex is shown in Fig. 2.

The Ni(II) complex crystallizes in the orthorhombic space group *Pbca*. The molecular structure consists of two Ni²⁺ and two (L)²⁻ units, and two coordinated pyridine molecules. The Ni1 and Ni1^{#1} atoms are both located in the N_2O_2 coordination cavity of the (L)²⁻ units. Each Ni atom is hexa-coordinated by two oxygen and two nitrogen atoms from an (L)²⁻ unit, one nitrogen atom of a coordinated pyridine molecule and one oxygen atom from another [Ni(L) (py)] moiety, and has a slightly distorted octahedral geometry (Fig. 2). In previous reports, the Salamo-type Ni(II) complexes are essentially mono- [30, 31], tri- [26, 31, 32] and hepta-nuclear [33], and the ratios of the ligand to Ni units are 1:1, 2:3 and 2:7. The Ni(II) complex reported here has the ratio 2:2.

3.6 Supramolecular interaction of the Ni(II) complex

In the crystal structure of the Ni(II) complex, there are two intramolecular hydrogen bonding interactions C9– H9B···O4 and C17–H17···O1 (Fig. 3) [41–44]. Selected bond lengths and angles for hydrogen bonding interactions are given in Table 3. As shown in Figs. 4 and 5, the molecules are connected by four intermolecular hydrogen bonding interactions which play a role



Fig. 5: View of the 2D supramolecular structure of the Ni(II) complex along the *bc* plane (hydrogen atoms, except those forming hydrogen bonds, are omitted for clarity).



Fig. 6: View of the 3D supramolecular structure of the Ni(II) complex (hydrogen atoms, except those forming hydrogen bonds, are omitted for clarity).

in constructing and stabilizing the crystal structure. Neighboring complex molecules are linked into an infinite 2D supramolecular network by intermolecular hydrogen bondings C20–H20···Cl1 and C17–H17···Br2. Finally, an infinite 3D supramolecular structure is formed (Fig. 6) [45, 46].

4 Conclusions

We have reported a new dinuclear Ni(II) complex, [{Ni(L) (C_5H_5N) }₂], with a new Salamo-type ligand H₂L. The Ni(II) complex contains two Ni²⁺ and two (L)²⁻ units, and two coordinated pyridine molecules. Each Ni(II) atom is hexa-coordinated by two oxygen and nitrogen atoms from an $(L)^{2-}$ unit, one nitrogen atom of the coordinated pyridine molecule and one oxygen atom from another [Ni(L)(py)] moiety. This structure is different from that of other Ni(II) complexes with the Salamo-type ligands reported early.

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