

2-(*N,N*-二羧甲基氨基甲基)氢醌金属配合物 (钴、镍、锌)的合成、晶体结构及电化学性质研究

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摘要: 合成了 3 个新的氢醌金属配合物: $[\text{Co}(\text{H}_2\text{cah})(\text{H}_2\text{O})_2] \cdot \text{H}_2\text{O}$ (**1**), $[\text{Ni}(\text{H}_2\text{cah})(\text{H}_2\text{O})_2] \cdot \text{H}_2\text{O}$ (**2**), $[\text{Zn}(\text{H}_2\text{cah})(\text{CH}_3\text{OH})(\text{H}_2\text{O})] \cdot \text{H}_2\text{O}$ (**3**) ($\text{H}_4\text{cah}=2-(N,N\text{-二羧甲基氨基甲基})\text{氢醌}$), 通过 X-射线单晶衍射, 红外光谱, 元素分析对它们进行了表征。这些配合物均为单核结构, 并且通过分子间的氢键形成了无限的超分子网状结构。电化学研究显示配合物 **1** 中的 $\text{Co}^{2+}/\text{Co}^{+}$ 对和配合物 **2** 中的 $\text{Ni}^{2+}/\text{Ni}^{+}$ 对的氧化还原是一个不可逆的过程, 但是氢醌到半醌之间的转换是一个准可逆的过程。

关键词: 钴(II)配合物; 镍(II)配合物; 锌(II)配合物; 晶体结构; 电化学

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Complexes of M^{II} ($\text{M}=\text{Co}, \text{Ni}, \text{Zn}$) with 2-[*N,N*-Bis(carboxymethyl) Aminomethyl] Hydroquinone: Syntheses, Crystal Structures and Electrochemical Properties

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Abstract: Three *p*-hydroquinone-metal complexes: $[\text{Co}(\text{H}_2\text{cah})(\text{H}_2\text{O})_2] \cdot \text{H}_2\text{O}$ (**1**), $[\text{Ni}(\text{H}_2\text{cah})(\text{H}_2\text{O})_2] \cdot \text{H}_2\text{O}$ (**2**) and $[\text{Zn}(\text{H}_2\text{cah})(\text{CH}_3\text{OH})(\text{H}_2\text{O})] \cdot \text{H}_2\text{O}$ (**3**), where $\text{H}_4\text{cah}=2-[N,N\text{-bis}(\text{carboxymethyl})\text{aminomethyl}]\text{hydroquinone}$, have been synthesized and characterized using X-ray diffraction single crystal structure determination, IR and elemental analysis. All these compounds were mononuclear structure, and complexes form an infinite supramolecular networks by intermolecular hydrogen bond. The cyclic voltametric analysis of **1** and **2** show that the electron transfer in the electrode reaction is irreversible for $\text{M}(\text{II})/\text{M}(\text{I})$ redox couples ($\text{M}=\text{Co}, \text{Ni}$) while reversible for process of interchange from *p*-hydroquinone to *p*-semiquinones. CCDC: 797009, **1**; 797010, **2**; 797011, **3**.

Key words: Co(II) complexe; Ni(II) complexe; Zn(II) complexe; crystal structure; electrochemistry

0 Introduction

Hydroquinone is a phenolic compound and it is a good general-purpose inhibitor of tyrosinase (one of the key enzymes in melanin synthesis), used as an intermediate in the synthesis of antioxidants and polymerization inhibitors^[1-2]. The three redox species of

the hydroquinone and hydroquinone derivatives, hydroquinone, *p*-semiquinone, and *p*-quinone, are important proton/organic electron sources and sinks, which play an essential role in the kinetics of related chemical systems and in electron/proton coupled biochemical processes^[3-5]. For instance, electron transfer reactions between *p*-quinone cofactors and transition metal

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centers are vital for all life, occurring in key biological processes as diverse as the oxidative maintenance of biological amine levels, tissue (collagen and elastin) formation, photosynthesis, and aerobic (mitochondrial) respiration^[6]. The metal ions in these systems lie in close proximity to *p*-semiquinone radicals resulting in immediate interaction^[4]. Moreover, the assembly of discrete molecular architectures through hydrogen bonds have attracted widespread scientific attention over recent years, the behavior of hydroquinone in a variety of hydrogen bond donating environments were explored by Warner and co-workers, which showed such hydrogen bonding networks^[7-9].

A strategy to prepare such species is to synthesize substituted, in the *o*-position, *p*-hydroquinones with substituents containing one or more donor atoms, thus enabling the metal atom to form chelating rings. In this work, using 2-[*N,N*-bis(carboxymethyl) aminomethyl] hydroquinone, we have synthesized three new Co^{II}, Ni^{II}, Zn^{II} complexes. The metal ions of the complexes were coordinated by the tetradentate H₂cah ligand. All these compounds were mononuclear structure, and extended supramolecular networks are constructed via secondary interactions such as hydrogen-bonding. Co^{II} and Ni^{II} complexes show that the electron transfer in the electrode reaction is irreversible for M(II)/M(I) redox couples (M=Co, Ni) while reversible for process of interchange from *p*-Hydroquinone to *p*-semiquinones.

1 Experimental

1.1 Materials and physical measurements

The ligand 2-[*N,N*-bis(carboxymethyl) aminomethyl] hydroquinone was prepared according to literature^[10]. All reagents and solvents for the synthesis and analysis were commercially available and used without further purification. IR spectra were taken on a Perkin-Elmer spectrum One FTIR spectrometer in the 4 000~400 cm⁻¹ region with KBr pellets. Elemental analyses (C, H, and N) were performed on an Elementar Vario EL analyzer. Electrochemical measurement was made at room temperature using a BAS Epsilon Electrochemical workstation with a conventional three-electrode cell consisting of a glassy carbon working, a

platinum wire auxiliary and a saturated calomel reference electrode (SCE). 0.1 mol·L⁻¹ KCl was used as supporting electrolytes in aqueous solution.

1.2 Synthesis of the [Co(H₂cah)(H₂O)₂]·H₂O (1)

A mixture of H₂cah (0.127 5 g, 0.5 mmol), Co(ClO₄)₂·6H₂O (0.183 0, 0.5 mmol) and Et₃N (0.5 mmol) was added into an methanol solution (15 mL). The pH value was adjusted to 5.0~6.0 by addition of NH₃·H₂O, the resulting solution was stirred for 2 h at room temperature and then filtered. Red block crystals were given by slow evaporation of the solvent for several days in a 77% yield. Anal. Calcd. for C₁₁H₁₇NO₉Co (%): C, 36.12; H, 5.02; N, 3.79; Found (%): C, 36.0; H, 5.13; N, 4.80. IR (KBr, cm⁻¹): 3 422.01 s, 3 260.32 m, 1 610.78 s, 1 585.55 s, 1 512.53 s, 1 457.92 w, 1 407.14 s, 1 326.45 m, 1 225.54 s, 977.06 m, 815.06 m, 623.64 w.

1.3 Synthesis of the [Ni(H₂cah)(H₂O)₂]·H₂O (2)

The same synthetic procedure as that for **1** was used except that Co(ClO₄)₂·6H₂O was replaced by Ni(NO₃)₂·6H₂O (0.145 4 g, 0.5 mmol), giving green black block crystals of complex **2** after several days. Anal. Calcd. for C₁₁H₁₇NO₉Ni(%): C, 36.10; H, 4.68; N, 4.52; Found(%): C, 35.06; H, 5.13; N, 4.79. IR (KBr, cm⁻¹): 3 424.05 s, 3 290.30 m, 1 613.25 s, 1 585.90 s, 1 512.53 s, 1 457.92 w, 1 407.54 s, 1 240.04 m, 1 208.62 s, 987.06 m, 817.04 m, 625.63 w.

1.4 Synthesis of the [Zn(H₂cah)(CH₃OH)(H₂O)]·H₂O (3)

The same synthetic procedure as that for **1** was used except that Co(ClO₄)₂·6H₂O was replaced by Zn(NO₃)₂·6H₂O (0.148 7 g, 0.5 mmol), giving colorless prism crystals of complex **3** after several days. Anal. Calcd for C₁₂H₁₉NO₉Zn(%): C, 37.37; H, 4.7; N, 3.63; Found(%): C, 36.98; H, 4.69; N, 3.72. IR (KBr, cm⁻¹): 3 420.04 s, 3 257.52 m, 1 610.79 s, 1 585.02 s, 1 513.26 s, 1 457.92 w, 1 405.56 s, 1 290.71 m, 1 213.13 s, 977.04 m, 814.03 m, 620.60 w.

1.5 X-ray crystallography

Single-crystal X-ray diffraction data for complexes **1**~**3** were collected on a Bruker Smart Apex II CCD diffractometer with Mo K α radiation (λ =0.071 073 nm) at room temperature. There was no evidence of crystal decay during data collection. The structures were

solved by the direct method and refined by the full-matrix least-squares method on F^2 using the SHELXTL 97 crystallographic software package (Table 1)^[11]. All non-hydrogen atoms were assigned anisotropically. The H atoms of the water molecules were located in a

difference Fourier map and the H atoms on all C atoms were positioned geometrically (C-H_{aromatic} 0.093 nm, C-H_{aliphatic} 0.097 nm). All the hydrogen atoms were refined with riding model.

CCDC: 797009, **1**; 797010, **2**; 797011, **3**.

Table 1 Crystallographic data and structure refinement details for complex **1**~**3**

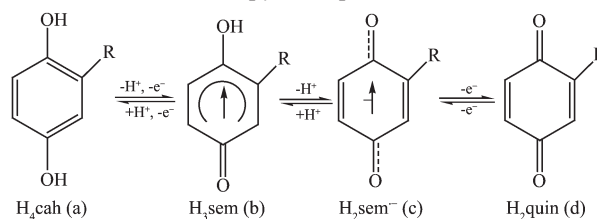
Complex	1	2	3
Empirical formula	C ₁₁ H ₁₇ CoNO ₉	C ₁₁ H ₁₇ NiNO ₉	C ₁₂ H ₁₉ ZnNO ₉
Formula weight	366.19	365.97	386.67
Crystal system	Monoclinic	Monoclinic	Monoclinic
Space group	$P2_1/c$	$P2_1/c$	$P2_1/c$
a / nm	0.741 03(13)	0.735 5	0.741 1(3)
b / nm	2.471 3(2)	2.483 8(9)	2.480 5(8)
c / nm	0.792 01(15)	0.788 0(3)	0.791 9(3)
β / (°)	99.94(3)	99.987(5)	100.014(4)
Volume / nm ³	1.428 6(4)	1.417 7(9)	1.433 6(8)
Z	4	4	4
Calculated density / (g·cm ⁻³)	1.703	1.715	1.787
$F(000)$	756	760	800
Crystal size / mm	0.45×0.42×0.37	0.42×0.38×0.35	0.49×0.38×0.34
θ range / (°)	1.65 to 25.00	1.64 to 25.01	1.64 to 25.01
Reflections collected	7 384	7 350	6 773
Independent reflections (R_{int})	2 516 (0.041 8)	2 502 (0.021 4)	2 299 (0.034 2)
Max. and min. transmission	0.655 9 to 0.604 2	0.637 0 to 0.587 7	0.585 9 to 0.479 2
Data / restraints / parameters	2 516 / 0 / 199	2 502 / 0 / 199	2 299 / 26 / 208
Goodness-of-fit on F^2	1.008	1.003	1.023
Final R indices ($I > 2\sigma(I)$)	$R_1=0.063$ 0, $wR_2=0.215$ 6	$R_1=0.026$ 5, $wR_2=0.065$ 1	$R_1=0.077$ 9, $wR_2=0.212$ 3
R indices (all data)	$R_1=0.077$ 3, $wR_2=0.227$ 6	$R_1=0.034$ 3, $wR_2=0.070$ 1	$R_1=0.089$ 6, $wR_2=0.225$ 5

2 Results and discussion

2.1 Structural description of **1**~**3**

Single crystal X-ray diffraction analysis reveals that crystal structure of complexes **1**~**3** are very similar, so only the structure of **1** is discussed detailedly herein. The selected bond distances and bond angles of complexes are given in Table 2. The anion of **1** (Fig.1) contains a bridging μ -*N,O,O,O*-*p*-hydroquinonate ligand (Scheme 1a), as it was clearly borne out by the observation: (i) the C-C bonds of the six-member ring in the ligand are very close to each other with a mean value of 0.138 5 nm; (ii) the C-O bond length (0.138 3(7)~0.138 5 (7) nm) is slightly longer than the bond length expected for *p*-hydroquinonates (0.135 4 nm)^[12-15]. Crystallographically independent Co^{II} ions displays a

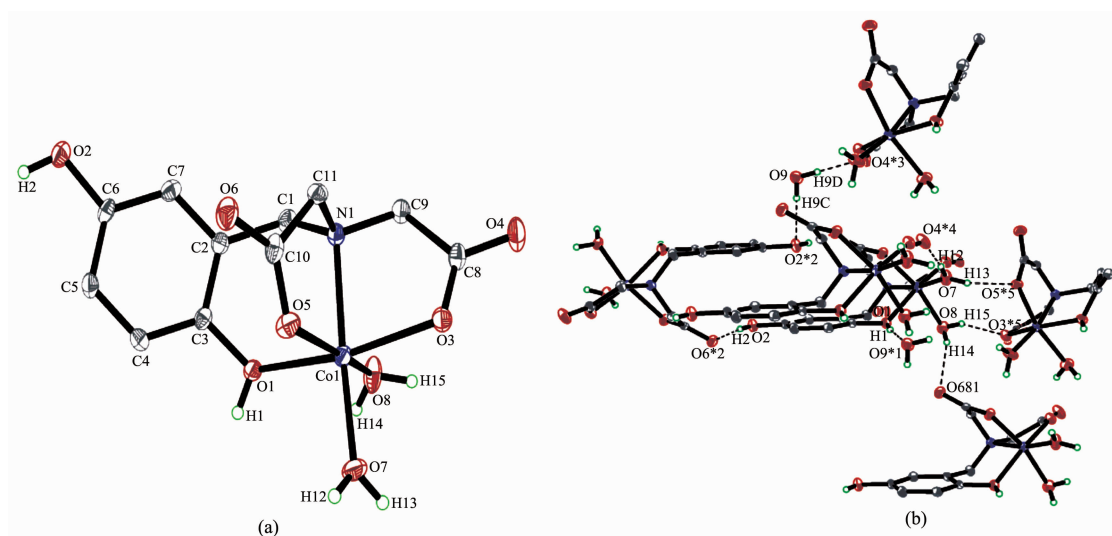
distorted octahedral NO₅ coordination environment, which is six-coordinated by one tetradentate H₂cah ligand and two coordinated water molecules (Fig.1a). The Co-N distance is 0.213 9(5) nm (Ni-N 0.207 5(2) nm; Zn-N 0.216 1(6) nm), while the Co-O distances are slightly shorter range from 0.202 0(5) to 0.211 6(4) nm (Ni-O distances from 0.201 3(2) to 0.209 0(2) nm; Zn-O distances from 0.201 2(6) to 0.216 9(5) nm). The best equatorial plane is defined by O1, O3, O5 and O8, while N1 and O7 occupy the apical sites. The mononu-



Scheme 1 Electron transfer in the *p*-hydroquinonate

Table 2 Selected bond lengths (nm) and bond angles ($^{\circ}$) for complex 1~3

1					
Co(1)-O(7)	0.202 0(5)	Co(1)-O(8)	0.208 2(5)	Co(1)-O(5)	0.207 6(5)
Co(1)-O(1)	0.211 6(4)	Co(1)-O(3)	0.208 1(5)	Co(1)-N(1)	0.213 9(5)
O(7)-Co(1)-O(5)	90.4(2)	O(3)-Co(1)-O(1)	162.86(19)	O(7)-Co(1)-O(3)	96.0(2)
O(8)-Co(1)-O(1)	80.4(2)	O(5)-Co(1)-O(3)	102.3(2)	O(7)-Co(1)-N(1)	169.2(2)
O(7)-Co(1)-O(8)	96.4(2)	O(5)-Co(1)-N(1)	81.76(2)	O(5)-Co(1)-O(8)	167.4(2)
O(3)-Co(1)-N(1)	78.44(19)	O(3)-Co(1)-O(8)	87.6(2)	O(8)-Co(1)-N(1)	92.7(2)
O(7)-Co(1)-O(1)	97.4(2)	O1-Co1-N1	89.94(19)	O(5)-Co(1)-O(1)	88.2(19)
2					
Ni(1)-O(8)	0.201 32(17)	Ni(1)-O(1)	0.204 58(16)	Ni(1)-O(3)	0.203 50(17)
Ni(1)-O(5)	0.209 01(16)	Ni(1)-O(7)	0.207 9(5)	Ni(1)-N(1)	0.207 52(19)
O(8)-Ni(1)-O(3)	90.05(7)	O(1)-Ni(1)-O(5)	167.27(6)	O(8)-Ni(1)-O(7)	94.15(7)
N(1)-Ni(1)-O(5)	91.42(7)	O(3)-Ni(1)-O(7)	170.27(7)	O(8)-Ni(1)-N(1)	172.19(7)
O(8)-Ni(1)-O(1)	96.11(7)	O(3)-Ni(1)-N(1)	83.93(7)	O(3)-Ni(1)-O(1)	99.54(7)
O(7)-Ni(1)-N(1)	92.57(7)	O(7)-Ni(1)-O(1)	88.75(7)	O(1)-Ni(1)-N(1)	80.05(7)
O(8)-Ni(1)-O(5)	93.44(7)	O(3)-Ni(1)-O(5)	88.83(7)	O(7)-Ni(1)-O(5)	82.17(6)
3					
Zn(1)-O(7)	0.201 2(6)	Zn(1)-O(3)	0.209 3(5)	Zn(1)-O(8)	0.206 3(6)
Zn(1)-O(1)	0.216 9(5)	Zn(1)-O(5)	0.208 8(5)	Zn(1)-N(1)	0.216 1(6)
O(7)-Zn(1)-O(8)	98.2(3)	O(3)-Zn(1)-O(1)	162.6(2)	O(7)-Zn(1)-O(5)	89.6(2)
N(1)-Zn(1)-O(1)	88.5(2)	O(8)-Zn(1)-O(5)	166.0(2)	O(7)-Zn(1)-N(1)	168.6(3)
O(7)-Zn(1)-O(3)	97.4(3)	O(8)-Zn(1)-N(1)	92.3(3)	O(8)-Zn(1)-O(3)	89.6(2)
O(5)-Zn(1)-N(1)	81.1(2)	O(5)-Zn(1)-O(3)	100.9(2)	O(3)-Zn(1)-N(1)	78.2(2)
O(7)-Zn(1)-O(1)	97.7(3)	O(5)-Zn(1)-O(1)	87.9(2)	O(8)-Zn(1)-O(1)	79.6(2)



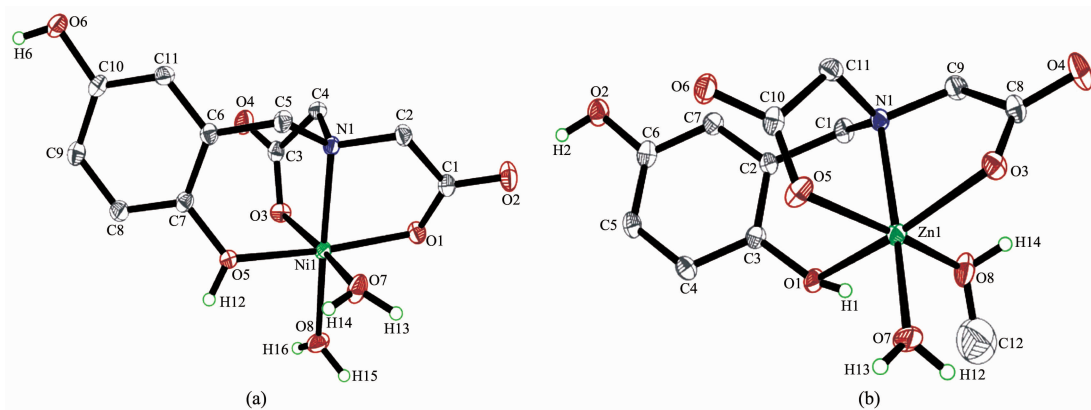
Uncoordinated H_2O molecules and part of the hydrogen atoms are omitted for clarity; Hydrogen bonds shown as dashed lines; symmetry codes: #1: $x, y, z+1$; #2: $-x+1, -y+2, -z+1$; #3: $x+1, -y+1/2, z-3/2$; #4: $x+1, y, z$; #5: $x, -y+1/2, z-1/2$

Fig.1 (a) Coordination environment of a mononuclear Co^{II} unit in complex 1; (b) Intermolecular hydrogen bonding in the complex 1

Table 3 Hydrogen bond lengths and bond angles for complex 1~3

D-H...A	$d(\text{D-H}) / \text{nm}$	$d(\text{H}\cdots\text{A}) / \text{nm}$	$d(\text{D}\cdots\text{A}) / \text{nm}$	$\angle \text{D-H}\cdots\text{A} / (^{\circ})$
1				
O(1)-H(1)...O(9#1)	0.082	0.179	0.260(7)	169
O(2)-H(2)...O(6#2)	0.082	0.195	0.260(7)	174
O(9)-H(9C)...O(2#2)	0.085	0.199	0.284(7)	179
O(9)-H(9D)...O(4#3)	0.085	0.194	0.279(7)	179
O(7)-H(12)...O(4#4)	0.085	0.187	0.271(8)	173
O(7)-H(13)...O(5#5)	0.085	0.207	0.286(7)	152
O(8)-H(14)...O(6#1)	0.085	0.212	0.278(7)	175
O(8)-H(15)...O(3#5)	0.085	0.191	0.269(7)	133
2				
O(6)-H(6)...O(4#1)	0.082	0.186	0.268(2)	178
O(5)-H(12)...O(9#2)	0.085	0.175	0.260(2)	173
O(7)-H(13)...O(1#3)	0.085	0.182	0.266(2)	169
O(7)-H(14)...O(4#2)	0.085	0.197	0.279(3)	163
O(8)-H(15)...O(3#3)	0.085	0.207	0.288(3)	159
O(8)-H(16)...O(2#4)	0.085	0.190	0.275(3)	178
O(9)-H(17)...O(6#1)	0.085	0.202	0.283(2)	158
O(9)-H(18)...O(2#4)	0.085	0.196	0.280(3)	170
3				
O(2)-H(2)...O(6#1)	0.082	0.188	0.270(8)	175
O(7)-H(12)...O(5#2)	0.085	0.214	0.286(9)	142
O(7)-H(13)...O(4#3)	0.085	0.188	0.270(9)	159
O(8)-H(14)...O(3#2)	0.085	0.217	0.268(8)	118
O(9)-H(16)...O(4#4)	0.088	0.199	0.282(9)	156
C(9)-H(9B)...O(9#5)	0.097	0.251	0.346(10)	165
C(12)-H(12A)...O(5#6)	0.096	0.238	0.296(17)	118
C(12)-H(12C)...O(7#2)	0.096	0.175	0.252(18)	135

Symmetry codes: #1: $x, y, z+1$; #2: $x+1, -y+2, -z+1$; #3: $x+1, -y+1/2, z-3/2$; #4: $x+1, y, z$; #5: $x, -y+1/2, z-1/2$ for **1**; #1: $x+1, y+1, z+1$; #2: $x, y, z-1$; #3: $x, y+3/2, z-1/2$; #4: $x-1, y, z$; #5: $x-1, y+3/2, z+1/2$ for **2**; #1: $x+1, y+2, z+1$; #2: $x, y+3/2, z+1/2$; #3: $x+1, y, z$; #4: $x+1, y+3/2, z-1/2$; #5: $x-1, y, z+1$; #6: $x, y, z+1$ for **3**.



Uncoordinated H_2O molecules and part of the hydrogen atoms are omitted for clarity

Fig.2 (a) Coordination environment of a mononuclear Ni^{II} unit in **2**; (b) Coordination environment of a mononuclear Zn^{II} unit in **3**

clear structure of the complex is similar to the hydroquinonate complexes of Mg(II)^[16], Cu(II)^[17], Fe(III)^[18], Cr(III)^[19], Ga(III)^[20] and Mo(VI)^[21].

In the complex **1**, two carboxyl groups are deprotonated in the H₂cch ligand, facilitating the formation of O(8)-H(14)···O(6#1), O(9)-H(9D)···O(4#3), O(7)-H(12)···O(4#4), O(7)-H(13)···O(5#5) and O(2)-H(2)···O(6#2) hydrogen bonds between water and phenolichydroxyl ligands. These hydrogen bonds are the most important intermolecular interaction in the crystal packing lead to the formation of the infinite supramolecular networks (Fig.1). And the supramolecular networks is also consolidated by H-bonding involving coordinated water, free water and phenolichydroxyl (O(8)-H(15)···O(3#5), O(9)-H(9C)···O(2#2), O(1)-H(1)···O(9#5)).

2.2 Cyclic voltammetry (CV) discussion

Cyclic voltammetry (CV) of **1** and **2** (Fig.3) were measured with a conventional three electrode cell in aqueous solution with complex condensation of 1.0×10^{-5} mol · L⁻¹. The measurement was carried out at room

temperature with scanning range of -1.500 ~ 1.750 V and scanning rate of 100 mV · s⁻¹. The CV curve of **1** shows one reversible redox peak observed at $E_{1/2} = 0.292$ V ($\Delta E_p = 106$ mV, $I_{pa}/I_{pc} = 0.97$) corresponding to the process of interchange from *p*-hydroquinone to *p*-semiquinones. The observation of a similar redox process corresponds to what were reported in other studies^[19,22]. Therefore, this also give further support to the semiquinonate and hydroquinonate redox levels of the organic ligand. However, the reductive response at -0.257 V which has no corresponding cathodic wave irreversibly is probably attributed to Co(II)/Co(I) redox couples^[23-24].

The CV of **2** shows that one reversible redox wave was observed at $E_{1/2} = 0.398$ V ($\Delta E_p = 57$ mV, $I_{pa}/I_{pc} = 0.94$). This reversible redox peak value is slightly more than the value of **1** but still in accordance with the process of interchange from *p*-hydroquinone to *p*-semiquinones. However, the reductive response at -0.164 V which has no corresponding cathodic wave irreversibly is probably due to Ni(II)/Ni(I) redox couples^[23-25].

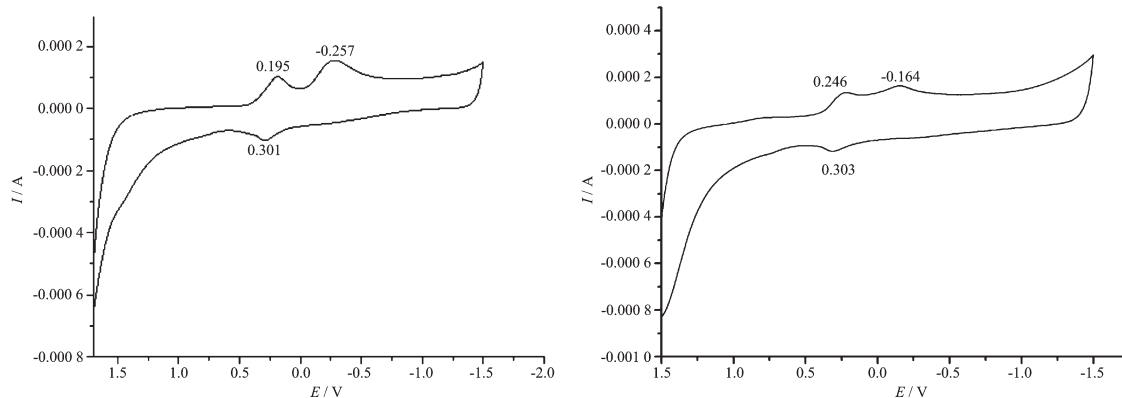


Fig.3 Cyclic voltammogram (CV) of complex **1** (left) and **2** (right)

3 Conclusions

In conclusion, we have synthesized and characterized three new *p*-Hydroquinone-metal complexes and these complexes were similarly mononuclear structure. They extended supramolecular networks are constructed via secondary interactions such as hydrogen-bonding. Especially, the cyclic voltametric analysis of Co^{II} and Ni^{II} complexes give further support to the semiquinonate and hydroquinonate redox levels of the organic ligand.

References:

- [1] Ran N Q, Knop D R, Draths K M, et al. *J. Am. Chem. Soc.*, **2001**,**123**:10927-10934
- [2] Kim G E, Lee J H, Jung S H. *J. Agric. Food Chem.*, **2010**,**58**: 9492-9497
- [3] Li N, Wang J C. *J. Phys. Chem. A*, **2008**,**112**:6281-6284
- [4] Drouza C, Keramidias A D. *Inorg. Chem.*, **2008**,**47**:7211-7224
- [5] Amorati R, Franchi P, Pedulli G F. *Angew. Chem.*, **2007**,**119**: 6452-6454
- [6] Costentin C, Robert M, Saveant J M. *Chem. Rev.*, **2010**,**110**:1-40

- [7] Feng X, Du H, Chen K, et al. *Cryst. Growth Des.*, **2010**, **10**(7):2901-2907
- [8] Cannon A S, Foxman B M, Guarrera D J, et al. *Cryst. Growth Des.*, **2005**, **5**(2):407-411
- [9] Saalfrank R W, Maid H, Scheurer A. *Angew. Chem.*, **2008**, **120**:8924-8956
- [10] FAN Neng-Ting(樊能廷). *Dictionary of Organic Synthesis*(有机合成词典). Beijing: Beijing Institute of Technology Press, **1992**.
- [11] (a) Sheldrick G M. *SHELXS-97, Program for X-ray Crystal Structure Solution*, Göttingen University, Germany, **1997**.
(b) Sheldrick G M. *SHELXL-97 Program for X-ray Crystal Structure Refinement*, Göttingen University, Germany, **1997**.
- [12] Chun H, Chaudhuri P, Weyhermüller T, et al. *Inorg. Chem.*, **2002**, **41**:790-795
- [13] Drouza C, Tolis V, Gramlich V, et al. *Chem. Commun.*, **2002**: 2786-2787
- [14] Foster C L, Liu X, Kilner C A, et al. *J. Chem. Soc. Dalton Trans.*, **2000**:4563-4568
- [15] Calderazzo F, Englert U, Pampaloni G, et al. *J. Chem. Soc. Dalton Trans.*, **2001**:2891-2898
- [16] Gao F, Meng B H, Wei Y B. *Acta Cryst.*, **2004**, **C60**:m360-362
- [17] Meng B H, Gao F, Du X Q. *Acta Cryst.*, **2004**, **E60**:m1103-1105
- [18] Li Y, Yung K F, Chan H S, et al. *Polyhedron*, **2001**, **20**:1687-1697
- [19] HUANG Fu-Ping(黄富平), WANG Yi-Xia(王仪夏), ZHAO Jing(赵婧), et al. *Chinese J. Inorg. Chem. (Wuji Huaxue Xuebao)*, **2008**, **24**(9):1523-1526
- [20] Olivier J, Florent M, Christian P, et al. *Eur. J. Inorg. Chem.*, **2005**:4417-4424
- [21] Snehadrinarayan K, Helen S E, Takunofi H, et al. *Inorg. Chem.*, **2006**, **45**:9619-9621
- [22] Petrangolini P, Alessandrini A, Berti L, et al. *J. Am. Chem. Soc.*, **2010**, **132**:7445-7453
- [23] Furutachi H, Okawa H. *Inorg. Chem.*, **1997**, **36**:3911-3918
- [24] JIAN Fang-Fang(建方方), XIAO Hai-Lian(肖海连), SUN Ping-Ping(孙萍萍). *Chinese J. Inorg. Chem. (Wuji Huaxue Xuebao)*, **2004**, **20**:1339-1343
- [25] Kianfara A H, Keramata L, Dostania M, et al. *Spectrochimica Acta: Part A*, **2010**, **77**:424-429