

两种 *N*-2-嘧啶基-4-氨基苯磺酰 Co(II)配合物的合成、 晶体结构及电催化性能研究

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摘要: 水热法合成两种新的 Co(II)化合物 $[\text{Co}(\text{SD})_2(2,2'\text{-bpy})]$ (**1**) 和 $\{[\text{Co}(\text{SD})_2(4,4'\text{-bpy})]\cdot 4\text{H}_2\text{O}\}_n$ (**2**) (SD=*N*-2-嘧啶基-4-氨基苯磺酰, 2,2'-bpy=2,2'-联吡啶, 4,4'-bpy=4,4'-联吡啶), 并通过元素分析、红外光谱、X 射线粉末衍射、热重分析、循环伏安法进行结构和性能表征。单晶 X-ray 衍射分析表明化合物 **1** 是三斜晶系, 空间群为 $P\bar{1}$; 化合物 **2** 是单斜晶系, 空间群为 $C2/c$ 。电催化实验发现化合物 **1** 和 **2** 对 H_2O_2 、HCHO 都具有良好的电催化活性。

关键词: 磺胺嘧啶; Co(II)配合物; 晶体结构; 电催化

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Syntheses, Crystal Structures and Electrochemical Behaviors of Two Co(II) Complexes with *N*-2-Pyrimidinyl-4-amino-benzenesulfonamides

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Abstract: Two new Co(II) complexes, $[\text{Co}(\text{SD})_2(2,2'\text{-bpy})]$ (**1**) and $\{[\text{Co}(\text{SD})_2(4,4'\text{-bpy})]\cdot 4\text{H}_2\text{O}\}_n$ (**2**) (SD=sulfadiazine, 2,2'-bpy=2,2'-bipyridine, 4,4'-bpy=4,4'-bipyridine) have been synthesized through hydrothermal reaction and characterized by elemental analysis, IR, powder X-ray diffraction (PXRD), thermogravimetric analysis (TGA) and cyclic voltammetry (CV). Single crystal X-ray analysis reveals that compound **1** crystallizes in the triclinic system, space group $P\bar{1}$ and compound **2** crystallizes in monoclinic crystal system, space group $C2/c$. It is found that these two compounds have electrochemical response behaviors for H_2O_2 and HCHO. CCDC: 977532, **1**; 977531, **2**.

Key words: sulfadiazine; Co(II) complexes; crystal structures; electrochemical behavior

Rapid development of coordination chemistry has been made in recent years not only for their potential applications in materials science but also for fascinating architectures, topologies and useful properties^[1-7]. The organic ligands containing appropriate functional groups in the molecule play important roles in the formation of coordination compounds and in the

stabilization of the whole structures^[8-9]. The heterocyclic-N donor ligands, especially pyridines, polypyridines and their substituted derivatives are important in generating of coordination complexes^[10-13].

A *N*-2-pyrimidinyl-4-amino-benzenesulfonamide (sulfadiazine), a kind of drug, is used widely in the prevention and cure of bacterial infections in humans

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because of its wide range, lower cost, and convenience to use^[14-15]. Previous studies found that some metal sulfanilamide complex activities was superior to free ligands or corresponding metal salts^[16-18]. The effectiveness of treatment seems to depend not only on the presence of a metal ion, but also crucially on the nature of the bonding point around of the metal ions^[19]. For example, zinc sulfadiazines^[20] are used to prevent bacterial infection in burned animals^[21-22] and silver sulfadiazines are used commercially for the treatment of topical burn^[23-24]. Otherwise, the crystal structures of metal-sulfadiazines with other organical ligands, such as pyridine^[25] and 5,5'-bimethyl-2,2'-bipyridine^[26] have also been reported.

In this paper, two cobalt sulfadiazine complexes with 2,2'-bipyridine (2,2'-bpy) or 4,4'-bipyridine (4,4'-bpy) were successfully synthesized and characterized by means of single crystal X-ray diffraction, TGA, powder crystal X-ray diffraction and elemental analysis. The electrochemical behaviors of the title complexes were studied via cyclic voltammetry.

1 Experimental

1.1 Materials and measurements

All chemicals were of analytical grade and used without further purification. Thermogravimetric analyses (TGA) were carried out from room temperature to 800 °C in air atmosphere using a STA449C integration thermal analyzer at a heating rate of 10 °C · min⁻¹. Powder X-ray diffraction (PXRD) data were collected on a Bruker D8 Focus X-ray diffractometer using Cu K α radiation. The calculated PXRD patterns were produced using the SHELXTL-XPOW program. Electrocatalytic experiments were carried out on a Model Epsilon Analyzer (BAS Inc., UAS) in a three-electrode cell: screen-printed carbon working electrode (SPCE) as the working electrode, platinum wire as the counter electrode, and Ag/AgCl/KCl as the reference electrode.

1.2 Synthesis of the complexes

1.2.1 Preparation of Co(SD)₂(2,2'-bpy) (1)

A mixture of [Co (OAc)₂] · 4H₂O (12.5 mg, 0.05 mmol), 2,2'-bpy (8.0 mg, 0.05 mmol), sulfadiazine

(12.5 mg, 0.05 mmol) and KOH (3.0 mg, 0.05 mmol) was placed in stainless steel pressure reaction kettle (25 mL) containing H₂O (10 mL). The reaction kettle was sealed, heated at 80 °C for 48 h, and allowed to cool to room temperature. Yield: 53% (based on Co). Elemental Anal. Calcd.(%) for **1** (C₃₀H₂₆CoN₁₀O₄S₂): C, 52.06; H, 4.21; N, 19.63; Found(%): C, 52.18; H, 4.11; N, 19.78. IR (KBr, cm⁻¹): 3 835(w), 3 452(vs), 2 064(w), 1 630(m), 1 597(m), 1 549(w), 1 500(w), 1 446(m), 1 421(s), 1 385(w), 1 287(w), 1 266(w), 1 139(m), 1 084(w), 1 012(w), 972(w), 831(w), 798(w), 771(w), 682(w), 584(m), 556(m), 466(w), 435(m).

1.2.2 Preparation of {[Co(SD)₂(4,4'-bpy)] · 4H₂O}_n (2)

A mixture of [Co(OAc)₂] · 4H₂O (12.5 mg, 0.05 mmol), 4,4'-bpy (10 mg, 0.05 mmol), sulfadiazine (12.5 mg, 0.05 mmol) and KOH (3.0 mg, 0.05 mmol) was placed in stainless steel pressure reaction kettle (25 mL) containing H₂O (10 mL). The reaction kettle was sealed, heated at 80 °C for 48 h, and allowed to cool to room temperature. Yield: 47% (based on Co). Elemental Anal. Calcd.(%) for **2** (C₃₀H₃₄CoN₁₀O₈S₂): C, 45.86; H, 4.36; N, 17.83; Found(%): C, 45.93; H, 4.28; N, 17.91. IR (KBr, cm⁻¹): 3 407 (vs), 3 221(s), 1 650(m), 1 595(m), 1 582(m), 1 557(w), 1 503(m), 1 446(m), 1 423(s), 1 413(w), 1 362(s), 1 315(s), 1 280(s), 1 261(s), 1 218(w), 1 131(m), 1 090(w), 1 015(w), 986(s), 840(w), 821(w), 803(w), 682(w), 637(w), 583(m), 551(m), 451(w), 434(m).

1.3 X-ray diffraction data collection and refinement

Single-crystal X-ray diffraction data were collected on a BRUKER SMART APEX II CCD diffractometer equipped with a graphite-monochromatic Mo K α radiation (λ =0.071 073 nm) using an ω scan mode at 293(2) K. Empirical absorption corrections were applied employing SADABS program^[27]. The structure was solved by direct methods and refined with a full-matrix least-squares technique using SHELXTL-97 program package^[28]. All hydrogen atoms of aromatic rings were placed geometrically and were subsequently refined in a riding-model approximation. All hydrogen atoms of the amidogen and H atoms of the guest water molecules in complex

2 were located from difference Fourier maps and refined with O-H distances restrained to 0.085 nm. All non-hydrogen atoms were refined anisotropically, and all hydrogen atoms were refined isotropically. The crystallographic data as well as details of the data

collection and refinement for complexes are listed in Table 1. Selected bond lengths and angles are given in Table 2 and Table 3.

CCDC: 977532, **1**; 977531, **2**.

Table 1 Crystal data and structure refinement for 1 and 2

Complex	1	2
Empirical formula	C ₃₀ H ₂₆ CoN ₁₀ O ₄ S ₂	C ₃₀ H ₃₄ CoN ₁₀ O ₈ S ₂
Formula weight	713.66	785.72
Temperature / K	293(2)	293(2)
Wavelength / nm	0.071 073	0.071 073
Crystal system	Triclinic	Monoclinic
Space group	<i>P</i> $\bar{1}$	<i>C</i> 2/ <i>c</i>
<i>a</i> / nm	1.028 4(3)	2.458 6(3)
<i>b</i> / nm	1.239 7(3)	1.135 63(16)
<i>c</i> / nm	1.287 4(3)	1.597 4(2)
α / (°)	75.518(3)	90
β / (°)	82.327(3)	128.693(10)
γ / (°)	73.545(3)	90
Volume / nm ³	1.520 6(6)	3.481 1(8)
<i>Z</i>	2	4
<i>D_c</i> / (g·cm ⁻³)	1.559	1.499
<i>F</i> (000)	734	1 628
Crystal size / mm	0.36×0.26×0.18	0.26×0.18×0.16
Theta range for data collection / (°)	1.64 to 27.46	2.08 to 27.50
Reflections collected / unique	13 437 / 6 802 (<i>R</i> _{int} =0.044 8)	14 745 / 3 989 (<i>R</i> _{int} =0.034 9)
Completeness to θ	97.8% (27.46°)	99.7 % (27.50°)
Refinement method	Full-matrix least-squares on <i>F</i> ²	Full-matrix least-squares on <i>F</i> ²
Data / restraints / parameters	6 802 / 0 / 431	3 989 / 4 / 241
Goodness-of-fit on <i>F</i> ²	1.003	1.254
Final <i>R</i> indices (<i>I</i> >2 σ (<i>I</i>))	<i>R</i> ₁ =0.047 0, <i>wR</i> ₂ =0.088 4	<i>R</i> ₁ =0.047 5, <i>wR</i> ₂ =0.159 5
<i>R</i> indices (all data)	<i>R</i> ₁ =0.105 5, <i>wR</i> ₂ =0.113 5	<i>R</i> ₁ =0.059 8, <i>wR</i> ₂ =0.170 7

Table 2 Selected bond lengths (nm) and angles (°)

Complex 1					
Co1-N2	0.211 0(3)	Co1-N4	0.214 7(3)	Co1-N6	0.219 4(3)
Co1-N7	0.213 8(3)	Co1-N1	0.213 9(3)	Co1-N5	0.222 5(3)
N2-Co1-N7	92.89(11)	N2-Co1-N6	145.83(11)	N1-Co1-N5	146.26(11)
N2-Co1-N1	76.42(11)	N1-Co1-N6	96.93(11)	N2-Co1-N4	117.01(11)
N1-Co1-N4	91.97(11)	N2-Co1-N5	97.28(10)	N6-Co1-N5	105.32(10)
N4-Co1-N5	60.97(10)	N7-Co1-N1	120.96(11)	N7-Co1-N6	61.44(10)
N4-Co1-N6	96.47(10)	N7-Co1-N4	140.73(11)	N7-Co1-N5	92.17(10)
Complex 2					
Co1-N1 ⁱⁱ	0.213 7(3)	Co1-N3	0.218 0(2)	Co1-N5	0.214 1(2)
Co1-N2	0.214 1(3)	Co1-N3 ^{iv}	0.218 0(2)	Co1-N5 ^{iv}	0.214 1(2)

Continued Table 2

N1 ⁱⁱ -Co1-N2	180.0(1)	N2-Co1-N3	85.57(6)	N5-Co1-N3	61.79(9)
N1 ⁱⁱ -Co1-N5 ^{iv}	89.35(6)	N5 ^{iv} -Co1-N3	118.33(9)	N1 ⁱ -Co1-N3 ^{iv}	94.43(6)
N2-Co1-N5 ^{iv}	90.65(6)	N5 ^{iv} -Co1-N5	178.69(11)	N2-Co1-N3 ^{iv}	85.57(6)
N1 ⁱⁱ -Co1-N5	89.35(6)	N1 ⁱⁱ -Co1-N3	94.43(6)	N5 ^{iv} -Co1-N3 ^{iv}	61.79(9)
N2-Co1-N5	90.65(6)	N5-Co1-N3 ^{iv}	118.33(9)	N3-Co1-N3 ^{iv}	171.14(11)

Symmetry codes: ⁱⁱ $x, y-1, z$; ^{iv} $-x+1, y, -z+1/2$.

Table 3 Hydrogen bond lengths and bond angles

D-H...A	$d(\text{D-H}) / \text{nm}$	$d(\text{H}\cdots\text{A}) / \text{nm}$	$d(\text{D}\cdots\text{A}) / \text{nm}$	$\angle \text{DHA} / (^\circ)$
Complex 1				
N9-H9B...N3 ^v	0.103	0.203	0.303 4(4)	165.2
N10-H10A...O2 ^{vi}	0.092(4)	0.207(5)	0.298 7(5)	175(4)
C4-H4...O3 ^{vii}	0.093	0.238	0.327 7(4)	162.9
C18-H18...O1 ^{vi}	0.093	0.231	0.320 4(4)	160.4
Complex 2				
N6-H6A...O4 ^{viii}	0.084(5)	0.231(5)	0.314(5)	171(4)
N6-H6B...N4 ^{ix}	0.080(5)	0.234(5)	0.309 6(5)	160(4)
O3-H3B...O3 ^x	0.092	0.23	0.268 3(16)	104.6
O4-H4A...O1	0.095	0.197	0.283(4)	148.7
O4-H4B...O2 ^{xi}	0.097	0.194	0.285 9(4)	157.1

Symmetry codes: ^v $2-x, -y, -z$; ^{vi} $1+x, y, z$; ^{vii} $1-x, 1-y, 1-z$; ^{viii} $1-x, -y, -z$; ^{ix} $1/2-x, -1/2+y, -1/2-z$; ^x $1/2-x, 3/2-y, -z$;^{xi} $1-x, 1-y, -z$.

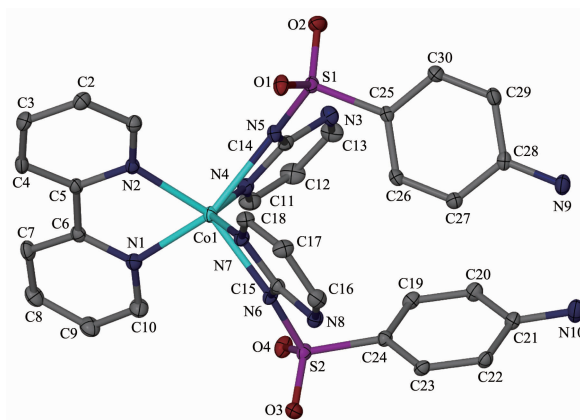
2 Results and discussion

2.1 Crystal structure

Crystal structure of complex **1**: Complex **1** is a mononuclear structure and crystallizes in the triclinic, space group $P\bar{1}$. The structural unit in **1** contains one Co(II) ion, two sulfadiazinate anions and one chelating 2,2'-bipyridine ligand. The central Co(II) ion has six-coordinated distorted octahedral geometry, of which two nitrogen atoms come from 2,2'-bipyridine and four nitrogen atoms come from sulfadiazinates (Fig.1). The coordination mode of Co(II) ion is similar to its manganese(II) complex [29]. The Co-N bond distances range from 0.211 0(3) to 0.222 5(3) nm in **1** (Table 2).

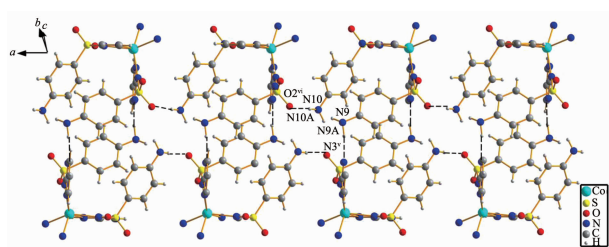
Different types of hydrogen bonds are observed in the structure of **1**: uncoordinated amino groups of sulfadiazinates act as hydrogen bonds donors, contributing hydrogen atoms to sulfonate oxygen atoms and nitrogen atoms, to form N9-H9B...N3^v, N10-H10A...O2^{vi} hydrogen bonding interactions, respectively ($d(\text{N9}\cdots\text{N3}^v)=0.303\ 4(4)$ nm, $\angle(\text{N9-H9B-N3}^v)=$

165.2°; $d(\text{N10}\cdots\text{O2}^{vi})=0.298\ 7(5)$ nm, $\angle(\text{N10-H10A-O2}^{vi})=175(4)^\circ$). The coordination units of **1** are further extended into a one-dimensional framework through the above classic hydrogen bonds interactions (Fig.2). The weak hydrogen bonding interactions were obtained in the structure of **1**: C4-H4...O3^{vii}, and C18-H18...O1^{vi} (Table 3).



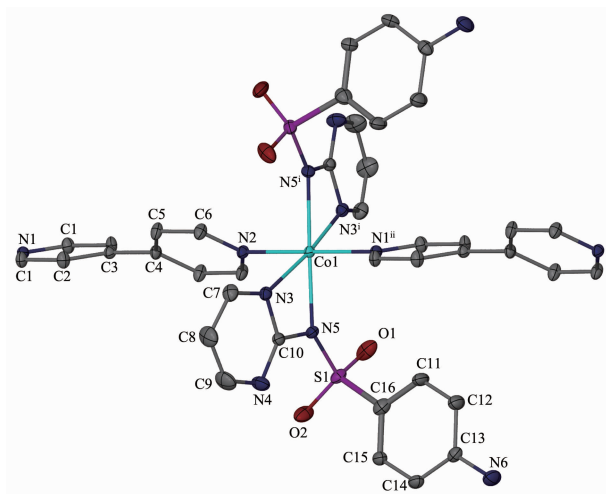
Atoms are represented by 20% probability displacement ellipsoids

Fig.1 Coordination environment of metal atom in compound **1**



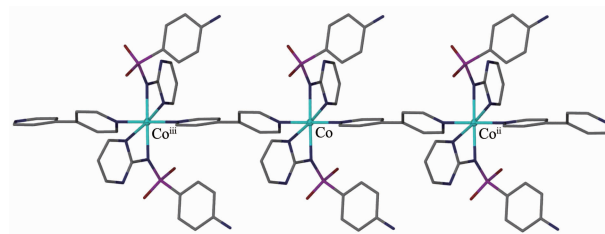
Some atoms are omitted for clarity; symmetry codes: ^v 2-x, -y, -z; ^{vi} 1+x, y, z

Structure description of complex **2**: Complex **2** crystallizes in monoclinic system with $C2/c$ space group. The coordination unit of the complex **1** consists of two sulfadiazine molecules, one cobalt ion and two 4,4'-bpy ligands. It's similar to the complex **1**, the central Co (II) ion adopts a six-coordinated mode, forming distorted octahedral geometry, of which four N atoms are from different sulfadiazine ligands, and other two are from the different 4,4'-bpy ligands (Fig. 3). In the crystal structure, 4,4'-bipyridine ligands act as bidentate bridging connectors to link Co(II) ions into a one-dimensional chain structure (Fig.4).

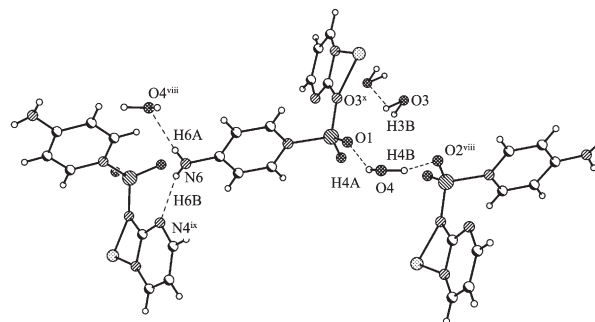


Atoms are represented by 35% probability displacement ellipsoids. Hydrogen atoms and guest water molecules are omitted for clarity; symmetry codes: ⁱ 1-x, y, 1/2-z; ⁱⁱ x, y-1, z

Within the crystal structure of complex **2**, the guest water molecule (O4) donates hydrogen atoms to sulfonate oxygen atoms (O1 and O2^{nj}) to form O4–H4A

Symmetry codes: ⁱⁱ $x, y-1, z$; ⁱⁱⁱ $x, 1+y, z$

$\cdots \text{O1}$ and $\text{O4}-\text{H4B} \cdots \text{O2}^{\text{xi}}$ strong hydrogen bonding interactions, respectively ($d(\text{O4} \cdots \text{O1})=0.283(4)$ nm, $\angle(\text{O4}-\text{H4A} \cdots \text{O1})=148.7^\circ$; $d(\text{O4} \cdots \text{O2}^{\text{xi}})=0.2859(4)$ nm, $\angle(\text{O4}-\text{H4B} \cdots \text{O2}^{\text{xi}})=157.1^\circ$). The uncoordinated amino group of sulfadiazine also acts as hydrogen bond donors, contributing hydrogen atoms to pyrimidine nitrogen atom and guest water oxygen atom to form $\text{N6}-\text{H6B} \cdots \text{O4}^{\text{viii}}$, $\text{N6}-\text{H6A} \cdots \text{N4}^{\text{ix}}$, respectively ($d(\text{N6} \cdots \text{O4}^{\text{viii}})=0.314(5)$ nm, $\angle(\text{N6}-\text{H6A} \cdots \text{O4}^{\text{viii}})=171(4)^\circ$; $d(\text{N6} \cdots \text{N4}^{\text{ix}})=0.3096(5)$ nm, $\angle(\text{N6}-\text{H6B} \cdots \text{N4}^{\text{ix}})=160(4)^\circ$) (Fig.5, Table 3). The structural units are extended into a three-dimensional framework through the intermolecular hydrogen bond and the offset face-to-face π - π stacking interactions of the neighboring pyrimidine rings with distances from 0.340 to 0.367 nm.



Symmetry codes: $\text{viii } 1-x, -y, -z; \text{ix } 1/2-x, -1/2+y, -1/2-z; \text{x } 1/2-x, 3/2-y, -z$

2.2 Powder X-ray diffraction analysis

In an attempt to confirm the homogeneity of the materials synthesized under the hydrothermal condition, we have analyzed the PXRD patterns of **1**, **2** and correlated the results with the simulated powder patterns obtained from the single crystal data. As depicted in Fig.6 and Fig.7, the basic features of the

PXRD patterns resemble the simulated ones from single crystal data for **1** and **2**, indicating that the bulk products obtained are homogenous in nature.

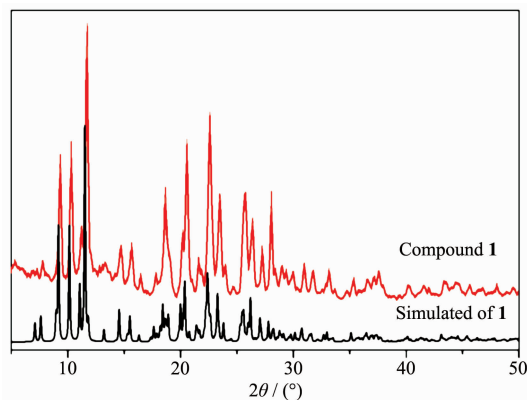


Fig.6 Experimental and simulated powder XRD patterns of **1**

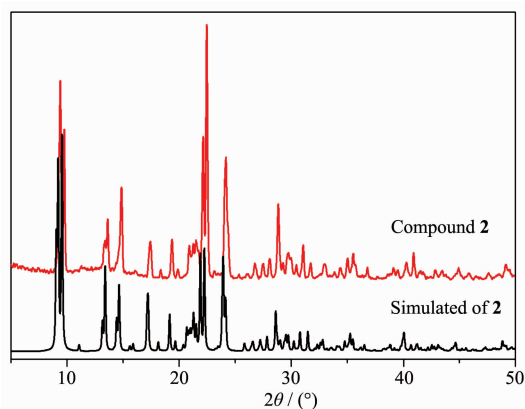


Fig.7 Experimental and simulated powder XRD patterns of **2**

2.3 Thermogravimetric analyses (TGA)

Thermogravimetric analysis (TGA) experiments were conducted to determine the thermal stability of the complexes, which is an important aspect for metal-organic frameworks^[30-32]. TGA was performed on crystalline samples of **1** and **2** under an air atmosphere from 20 to 800 °C. In complex **1**, the first sharp drop of weight was observed started from 240 and ended to 500 °C, with a weight loss of 54.7% (Calcd. 56.6%) corresponding to one sulfadiazine and one 2,2'-bipyridine molecule per formula unit $[\text{Co}(\text{SD})_2(2,2'\text{-bpy})]$, which indicated the stability of the framework of **1** up to 240 °C. The second sharp weight change of 34.3% (Calcd. 34.9%) from 500 to 570 °C, assigned to the other sulfadiazine ligands. In the complex **2**, the weight loss of 28.5 % (Calcd. 29.1%) corresponded to

one 4,4'-bipyridine molecule and four guest water molecules per formula unit $\{[\text{Co}(\text{SD})_2(4,4'\text{-bpy})]\cdot 4\text{H}_2\text{O}\}_n$ from 100 to 370 °C. The subsequent weight loss was 61.8% (Calcd. 63.4%) from 370 to 620 °C, assigned to the other sulfadiazine ligands.

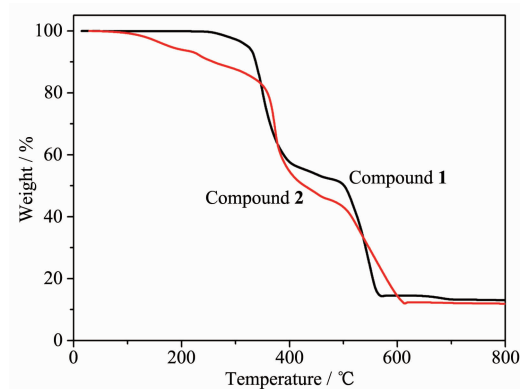


Fig.8 TGA curves of the title complex **1** and **2**

2.4 Catalysis behavior of Compounds

Cyclic voltammetry (CV) is a valuable and convenient tool for providing electrocatalytic information regarding the modified electrodes, as well as the activity and stability of immobilized reagents^[33-34]. Here, the KH_2PO_4 buffer solution (PBS, $C=0.1 \text{ mol}\cdot\text{L}^{-1}$, $\text{pH}=7.0$) was prepared by adding the NaOH aqueous solution ($3.0 \text{ mmol}\cdot\text{L}^{-1}$), in which the CV experiments were carried out at a scan rate of $50 \text{ mV}\cdot\text{s}^{-1}$. The cyclic voltammograms of the electrodes in different environments are shown in Fig.9 to Fig.12.

The bare screen-printed carbon working electrode (SPCE) in PBS exhibited the anodic peak of H_2O_2 at -0.93 V (Fig.9b) and -0.88 V (Fig.10b) after adding a

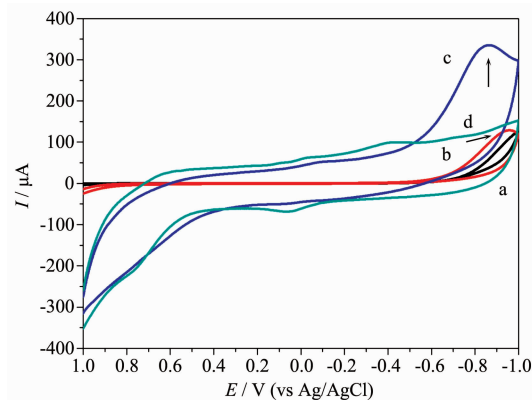


Fig.9 CVs in PBS solution ($\text{pH}=7.0$): (a) Bare SPCE; (b) SPCE after adding H_2O_2 ; (c) SPCE/Sample of **1** after adding H_2O_2 ; (d) SPCE/Sample of **1** without adding H_2O_2

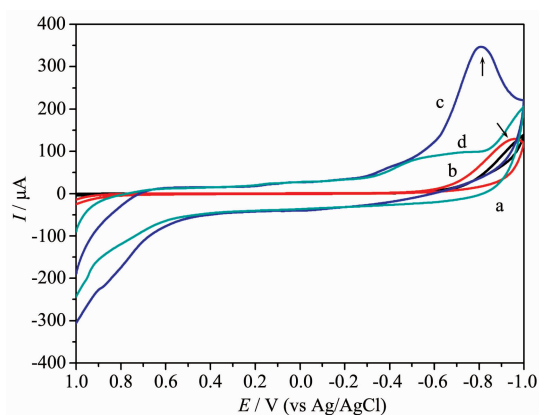


Fig.10 CVs in PBS solution (pH=7.0): (a) Bare SPCE; (b) SPCE after adding H_2O_2 ; (c) SPCE/Sample of **2** after adding H_2O_2 ; (d) SPCE/Sample of **2** without adding H_2O_2

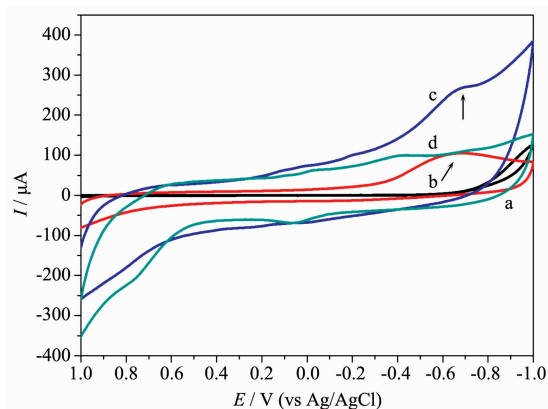


Fig.11 CVs in PBS solution (pH=7.0): (a) Bare SPCE; (b) SPCE after adding HCHO; (c) SPCE/Sample of **1** after adding HCHO; (d) SPCE/Sample of **1** without adding HCHO

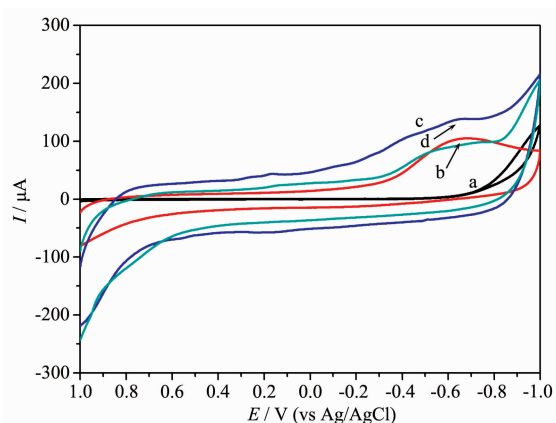


Fig.12 CVs in PBS solution (pH=7.0): (a) Bare SPCE; (b) SPCE after adding HCHO; (c) SPCE/Sample of **2** after adding HCHO; (d) SPCE/Sample of **2** without adding HCHO

H_2O_2 solution of $5.0 \text{ mmol} \cdot \text{L}^{-1}$. Comparing with the modified electrodes of SPCE/Sample of **1** and **2**

without adding H_2O_2 (Fig.9d, Fig.10d), the modified electrodes of the SPCE/Samples of **1** and **2** after adding H_2O_2 showed that the anodic current increased noticeably while the cathodic current decreased (Fig. 9c, Fig.10c), respectively, which indicated that both samples **1** and **2** had an electrocatalytic response to H_2O_2 , resulting from the reduction reaction of H_2O_2 to H_2O . Similar electrocatalytic experiments to HCHO for compound **1** and **2** was performed. The modified electrodes of the SPCE/Samples of **1** and **2** showed that the anodic current increased noticeably while the cathodic current decreased (Fig.11c, Fig.12c), which indicated that samples **1** and **2** had an electrocatalytic response to HCHO, resulting from the reduction reaction of HCHO to CH_3OH .

3 Conclusions

In this work, two new cobalt-sulfadiazine complexes have been synthesized and characterized. TGA analyses show high thermal stability of the frameworks for **1**. CV indicates that the compounds **1** and **2** have electrocatalytic activities towards H_2O_2 and HCHO. This work may provide useful information to assemble new functional materials.

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