

# Synthesis, Crystal Structure, Properties and Thermoanalysis of Complexes of Cu(II) and Ni(II) with Taurine-5-chlorosalicylaldehyde Schiff Base

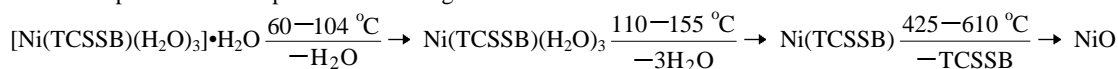
ZHANG, Shu-Hua<sup>a,b</sup>(张淑华)    JIANG, Yi-Min<sup>\*b</sup>(蒋毅民)    ZHOU, Zhong-Yuan<sup>c</sup>(周忠远)

<sup>a</sup> Department of Material and Chemical Engineering, Guilin University of Technology, Guilin, Guangxi, 541004, China

<sup>b</sup> College of Chemistry and Chemical Engineering, Guangxi Normal University, Guilin 541004, China

<sup>c</sup> Department of Applied Biology and Chemical Technology, Hongkong Polytechnic University, Hongkong, China

The reactions of transition metal salts with taurine 5-chlorosalicylaldehyde Schiff base gave two complexes [Ni(TCSSB)(H<sub>2</sub>O)<sub>3</sub>]•H<sub>2</sub>O (**1**) and [Cu(TCSSB)(H<sub>2</sub>O)<sub>2</sub>]<sub>2</sub>•[Cu(TCSSB)<sub>2</sub>]•6H<sub>2</sub>O (**2**) (TCSSB=taurine-5-chlorosalicylaldehyde Schiff base), which were characterized by elemental analysis and X-ray diffraction analysis. The complex **1** crystallized in monoclinic system with space group *P2<sub>1</sub>/c*, and *a*=1.4816(2) nm, *b*=1.3953(2) nm, *c*=0.7466(1) nm, *β*=100.499(3)°, *V*=1.5176(4) nm<sup>3</sup>, *Z*=4, and an infinite 3-D network structure was formed by hydrogen bonds among sulfo group, crystal water and coordinated water. Complex **2** crystallized in triclinic system with space group *P1̄*, with the cell parameters: *a*=0.6413(2) nm, *b*=1.4596(3) nm, *c*=1.6188(4) nm, *α*=102.473(5)°, *β*=98.979(4)°, *γ*=101.739°, *V*=1.4165(6) nm<sup>3</sup>, *Z*=1. The coordination environment between Cu(1) and Cu(2) is different. Cu(1) is slightly distorted square pyramidal while Cu(2) is distorted square-plane. The complex **1** is mononuclear while the complex **2** is made up of two coordinated subunits, namely [Cu(TCSSB)<sub>2</sub>] and [Cu(TCSSB)(H<sub>2</sub>O)<sub>2</sub>]<sub>2</sub>. Besides that the TG-DTG of the complex **1** was analyzed, the thermal decomposition reaction of the complex was studied under a non-isothermal condition by TG-DTG. The TG and DTG curves indicate that the complex was decomposed in three stages:



**Keywords**    molecular structure, taurine-5-chlorosalicylaldehyde Schiff base, copper(II), nickel(II), TG-DTG

## Introduction

The complexes of amino acid Schiff base have received considerable attention because of the interests in the biological field.<sup>1-4</sup> Taurine is a special amino acid in human beings. Recently, the complexes of taurine condensation salicylaldehyde Schiff base have been reported,<sup>3-7</sup> but the complexes of taurine condensation derived salicylaldehyde Schiff base have not been reported yet.

In order to examine its anticancer and antibiosis activity of the complexes of taurine condensation derived salicylaldehyde Schiff base, we synthesized two complexes of taurine 5-chlorosalicylaldehyde Schiff base.

## Experimental

### Materials

All solvent and chemicals were commercial reagents and were used without further purification. 5-Chloro-

salicylaldehyde was synthesized according to reference.<sup>8</sup>

### Physical measurements

The elemental analysis was performed on a PE 1700 CHN auto elemental analyzer. The crystal structure was determined by single-crystal X-ray diffraction and SHELXL crystallographic software of molecular structure. The TG analysis was performed on TG209.

### Syntheses

**Synthesis of complex 1:** Complex **1** was prepared by mixing an ethanol-water solution of 5-chlorosalicylaldehyde (1 mmol), taurine (1 mmol), potassium hydrate (1 mmol) with heating stirring. After two hours, an aqueous solution containing 1 mmol of Ni(AcO)<sub>2</sub>•4H<sub>2</sub>O was added dropwise under stirring. The pH value of the mixture was adjusted to 5—6 with 0.5 mol/L HCl solution. The resulting solution was left at room temperature, green crystals of the complex **1** were obtained after 25 d.

\* E-mail: zsh720108@21cn.com

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Found C 27.48, H 4.18, N 3.55, S 8.14;  $C_9H_{16}ClNNiO_8S$  requires C 27.54, H 4.11, N 3.57, S 8.17.

**Synthesis of complex 2:** Complex 2 was prepared as described above except that  $Ni(AcO)_2 \cdot 4H_2O$  was used instead of  $Cu(AcO)_2 \cdot 4H_2O$ , with slow evaporation of the reaction mixture. Good quality blue crystal was yielded in about two weeks. Found C 30.42, H 3.54, N 3.78, S 9.11;  $C_{36}H_{52}Cl_4Cu_3N_4O_{26}S_4$  requires C 30.50, H 3.52, N 3.70, S 9.05.

### Structure determination

The data of the two complexes were collected on a Bruker CCD area detector diffractometer equipped with a graphite-monochromatized  $Mo\ K\alpha$  radiation ( $\lambda = 0.071073\text{ nm}$ ) at 294(2) K. The structures were solved by direct methods and succeeding difference Fourier synthesis, and then refined by full-matrix least-squares techniques anisotropically for all non-hydrogen atoms. All hydrogen atoms were added according to theoretical modes. All calculations were performed by using SHELXL-97 program package.<sup>9</sup>

**Compound 1:** A green crystal of dimension 0.32 mm  $\times$  0.16 mm  $\times$  0.10 mm was chosen for the measurement. A total of 10101 reflections were collected in the range of  $3.14^\circ \leq \theta \leq 27.53^\circ$  using  $\omega$ - $\theta$  scan technique, of which 3475 ( $R_{int} = 0.0511$ ) were unique. The final convergence indices  $R = 0.0440$  and  $wR = 0.0823$  ( $w = 1/[S^2(F_o^2) + (0.0300P)^2 + 0.0000P]$  where  $P = (F_o^2 + 2F_c^2)/3$ ),  $S = 0.980$ , and  $(\Delta/\sigma)_{max} = 0.001$ . The maximum peak in the final difference Fourier map is  $473\text{ e}\cdot\text{nm}^{-3}$  and the minimum peak,  $-469\text{ e}\cdot\text{nm}^{-3}$ .

**Compound 2:** A blue crystal of dimension 0.40 mm  $\times$  0.32 mm  $\times$  0.28 mm was chosen for the measurement. A total of 9581 reflections were collected in the range of  $2.67^\circ \leq \theta \leq 27.54^\circ$  using  $\omega$ - $\theta$  scan technique of which 6402 ( $R_{int} = 0.0214$ ) were unique. The final convergence indices  $R = 0.0453$  and  $wR = 0.1158$  ( $w = 1/[S^2(F_o^2) + (0.0650P)^2 + 0.0000P]$  where  $P = (F_o^2 + 2F_c^2)/3$ ),  $S = 1.021$ , and  $(\Delta/\sigma)_{max} = 0.000$ . The maximum and the minimum peak values in the final difference Fourier map are 931 and  $-441\text{ e}\cdot\text{nm}^{-3}$ , respectively.

## Results and discussion

Selected bond distances and angles are listed in Tables 1 and 2, and fractional atomic coordinates and equivalent isotropic thermal parameters for non-hydrogen atoms are summarized in Tables 3 and 4. The molecular structures of the title complexes with the atom numbering scheme are illustrated in Figures 1 and 2, the packing drawings of the title complexes are illustrated in Figures 3 and 4.

### Structure of $[Ni(TCSSB)(H_2O)_3] \cdot H_2O$ (1)

The complex 1 is mononuclear. The nickel(II) cation is coordinated by a nitrogen and two oxygen atoms from taurine 5-chlorosalicylaldehyde Schiff base and three oxygen atoms from three water molecules to form a slightly distorted octahedron with oxygen atoms

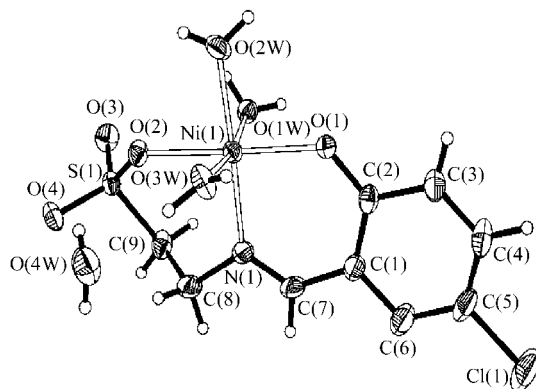


Figure 1 Molecular structure of the complex 1.

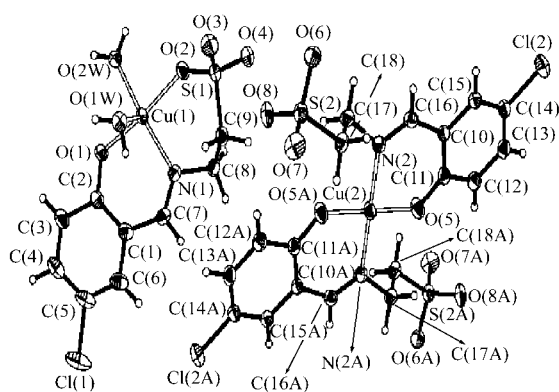


Figure 2 Molecular structure of the complex 2 (drawing 50% of  $[Cu(TCSSB)(H_2O)_2]_2$ ).

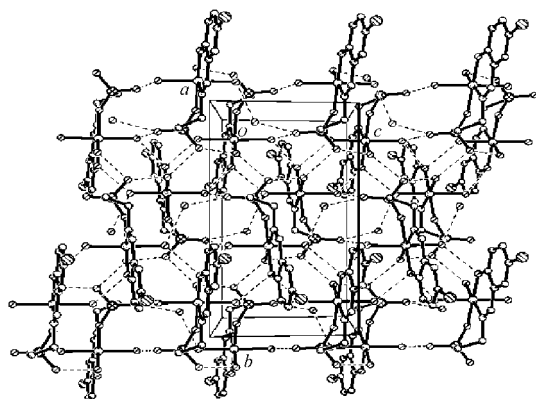


Figure 3 Packing drawing of the complex 1.

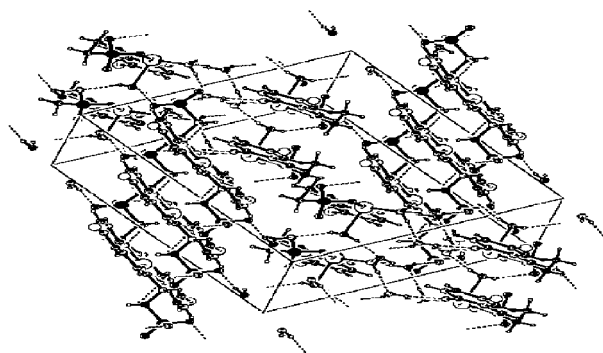


Figure 4 Packing drawing of the complex 2.

**Table 1** Selected bond distances ( $\times 10^{-1}$  nm) and bond angles ( $^{\circ}$ ) of the complex **1**<sup>a</sup>

Ni(1)—O(1)	2.003(2)	Ni(1)—N(1)	2.050(3)	Ni(1)—O(2W)	2.067(2)
Ni(1)—O(3W)	2.036(2)	Ni(1)—O(1W)	2.070(2)	Ni(1)—O(2)	2.079(2)
S(1)—O(4)	1.450(2)	S(1)—O(3)	1.453(2)	S(1)—O(2)	1.459(2)
N(1)—C(7)	1.273(4)	N(1)—C(8)	1.473(4)	Cl(1)—C(5)	1.749(4)
O(1)-Ni(1)-O(3W)	92.51(9)	O(1)-Ni(1)-N(1)	90.92(10)	O(3W)-Ni(1)-N(1)	93.18(11)
O(1)-Ni(1)-O(2W)	90.20(9)	O(3W)-Ni(1)-O(2W)	82.76(10)	N(1)-Ni(1)-O(2W)	175.84(11)
O(1)-Ni(1)-O(1W)	93.64(9)	O(3W)-Ni(1)-O(1W)	169.63(9)	N(1)-Ni(1)-O(1W)	95.06(10)
O(2W)-Ni(1)-O(1W)	88.87(9)	O(1)-Ni(1)-O(2)	177.86(9)	O(2W)-Ni(1)-O(2)	87.72(9)
O(3W)-Ni(1)-O(2)	86.75(9)	N(1)-Ni(1)-O(2)	91.13(10)	O(1W)-Ni(1)-O(2)	86.81(9)
O(4)-S(1)-O(3)	112.36(15)	O(4)-S(1)-O(2)	111.19(14)	O(3)-S(1)-O(2)	113.27(14)
O(4)-S(1)-C(9)	107.29(16)	O(3)-S(1)-C(9)	106.17(17)	O(2)-S(1)-C(9)	106.02(14)

<sup>a</sup> Symmetry transformations used to generate equivalent atoms: #1  $-x+1, -y, -z+1$ .**Table 2** Selected bond distances ( $\times 10^{-1}$  nm) and bond angles ( $^{\circ}$ ) of the complex **2**<sup>a</sup>

Cu(1)—O(1)	1.902(2)	S(1)—O(2)	1.471(3)	S(2)—O(6)	1.456(3)
Cu(1)—O(2W)	1.961(3)	Cu(2)—O(5)#1	1.889(3)	S(2)—C(18)	1.763(4)
Cu(1)—N(1)	1.974(3)	Cu(2)—O(5)	1.889(3)	O(5)—C(11)	1.307(4)
Cu(1)—O(2)	1.981(3)	Cu(2)—N(2)	2.018(3)	N(2)—C(16)	1.287(4)
Cu(1)—O(1W)	2.413(3)	Cu(2)—N(2)#1	2.018(3)	N(2)—C(17)	1.485(4)
S(1)—O(3)	1.439(3)	S(2)—O(7)	1.453(3)	N(1)—C(7)	1.278(4)
S(1)—O(4)	1.455(3)	S(2)—O(8)	1.453(3)	N(1)—C(8)	1.463(5)
O(1)-Cu(1)-O(2W)	87.02(11)	N(1)-Cu(1)-O(2)	92.33(12)	O(5)#1-Cu(2)-O(5)	180.0
O(1)-Cu(1)-N(1)	94.13(12)	O(1)-Cu(1)-O(1W)	90.06(11)	O(5)#1-Cu(2)-N(2)	88.13(11)
O(2W)-Cu(1)-N(1)	166.73(12)	O(2W)-Cu(1)-O(1W)	99.71(11)	O(5)-Cu(2)-N(2)	91.87(12)
O(1)-Cu(1)-O(2)	171.56(11)	N(1)-Cu(1)-O(1W)	93.51(11)	O(5)#1-Cu(2)-N(2)#1	91.87(12)
O(2W)-Cu(1)-O(2)	85.45(11)	O(2)-Cu(1)-O(1W)	94.96(10)	O(5)-Cu(2)-N(2)#1	88.13(11)
N(2)-Cu(2)-N(2)#1	180.00(14)	N(1)-Cu(1)-O(2)	92.33(12)	O(5)#1-Cu(2)-O(5)	180.0

<sup>a</sup> Symmetry transformations used to generate equivalent atoms: #1  $-x+1, -y, -z+1$ .**Table 3** Non-hydrogen fractional atomic coordinate ( $\times 10^4$ ) and equivalent isotropic temperature factors ( $\text{nm}^2 \times 10$ ) of the complex **1**

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{eq}}^a$	Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{eq}}^a$
Ni(1)	6547(1)	6225(1)	3821(1)	27(1)	N(1)	7765(2)	5490(2)	4232(4)	37(1)
Cl(1)	11146(1)	8350(1)	6081(2)	104(1)	C(1)	8768(2)	6897(2)	4808(5)	32(1)
S(1)	5970(1)	4052(1)	2694(1)	30(1)	C(2)	8102(2)	7632(2)	4526(4)	44(1)
O(1W)	6378(2)	6214(2)	1007(3)	33(1)	C(3)	8421(3)	8584(3)	4756(5)	57(1)
O(2W)	5309(2)	6938(2)	3597(3)	41(1)	C(4)	9331(3)	8798(3)	5243(6)	58(1)
O(3W)	6462(2)	6201(2)	6512(3)	43(1)	C(5)	9979(3)	8068(3)	5496(6)	55(1)
O(4W)	6376(2)	4404(2)	7859(3)	58(1)	C(6)	9700(3)	7144(3)	5298(6)	42(1)
O(1)	7216(1)	7479(1)	4062(3)	31(1)	C(7)	8547(2)	5890(3)	4642(5)	49(1)
O(2)	5809(1)	4949(1)	3587(3)	33(1)	C(8)	7747(2)	4434(2)	4220(6)	41(1)
O(3)	5469(2)	3984(2)	835(3)	45(1)	C(9)	7152(2)	4048(2)	2547(5)	37(1)
O(4)	5810(2)	3233(2)	3791(3)	42(1)					

<sup>a</sup>  $U_{\text{eq}}$  is defined as one third of the trace of the orthogonalized  $U_{ij}$  tensor.

**Table 4** Non-hydrogen fractional atomic coordinate ( $\times 10^4$ ) and equivalent isotropic temperature factors ( $\text{nm}^2 \times 10$ ) of the complex **2**

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{eq}}^a$	Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{eq}}^a$
Cu(1)	8571(1)	3738(1)	1611(1)	32(1)	Cl(2)	-5615(2)	-2834(1)	105(1)	63(1)
Cl(1)	19476(2)	5697(1)	4183(1)	81(1)	S(2)	5499(2)	-1632(1)	1859(1)	35(1)
S(1)	5027(1)	1871(1)	1592(1)	33(1)	O(5)	3089(4)	-281(2)	5739(2)	46(1)
O(1W)	10232(4)	2575(2)	836(2)	43(1)	O(6)	3502(5)	-2149(2)	1227(2)	47(1)
O(2W)	7252(4)	4191(2)	647(2)	41(1)	O(7)	7208(5)	-2150(2)	1832(2)	57(1)
O(1)	11105(4)	4739(2)	1736(2)	42(1)	O(8)	6159(5)	-640(2)	1798(2)	47(1)
O(2)	5694(4)	2819(2)	1407(2)	43(1)	N(2)	2839(5)	-888(2)	3950(2)	32(1)
O(3)	4805(5)	1075(2)	856(2)	50(1)	C(10)	104(5)	-1452(2)	4736(2)	29(1)
O(4)	3077(4)	1826(2)	1952(2)	44(1)	C(11)	1127(6)	-845(3)	5567(2)	31(1)
N(1)	9547(5)	3477(2)	2737(2)	32(1)	C(12)	-24(6)	-857(3)	6237(2)	37(1)
C(1)	13228(6)	4526(3)	3003(3)	38(1)	C(13)	-2079(6)	-1458(3)	6104(3)	38(1)
C(2)	12989(6)	4899(3)	2284(3)	37(1)	C(14)	-3015(6)	-2068(3)	5289(3)	38(1)
C(3)	14847(6)	5499(3)	2143(3)	48(1)	C(15)	-1976(6)	-2068(3)	4617(3)	35(1)
C(4)	16828(7)	5726(3)	2705(3)	55(1)	C(16)	1006(6)	-1423(3)	3982(2)	33(1)
C(5)	16997(7)	5374(3)	3445(3)	52(1)	C(17)	3264(6)	-942(3)	3066(2)	34(1)
C(6)	15253(6)	4781(3)	3587(3)	47(1)	C(18)	4856(6)	-1569(3)	2886(2)	36(1)
C(7)	11456(6)	3857(3)	3204(2)	37(1)	O(3W)	10802(4)	6015(2)	729(2)	45(1)
C(8)	8064(6)	2824(3)	3077(2)	37(1)	O(4W)	7076(4)	-3405(2)	198(2)	49(1)
C(9)	7133(6)	1838(3)	2415(3)	38(1)	O(5W)	-348(5)	90(2)	1057(2)	60(1)
Cu(2)	5000	0	5000	33(1)					

<sup>a</sup>  $U_{\text{eq}}$  is defined as one third of the trace of the orthogonalized  $U_{ij}$  tensor.

[O(1W), O(3W)] of water at apical positions. The total angle of O(1)-Ni(1)-N(1) ( $90.92^\circ$ ), O(1)-Ni(1)-O(2W) ( $90.20^\circ$ ), N(1)-Ni(1)-O(2) ( $91.13^\circ$ ), and O(2)-Ni(1)-O(2W) ( $87.72^\circ$ ) is  $359.97^\circ$  (nearly  $360^\circ$ ). That means that O(1), N(1), O(2W), O(2), Ni(1) atoms belong to a plane nearly. The interatomic distances of Ni(1)—O(1), Ni(1)—O(3W), Ni(1)—N(1), Ni(1)—O(2W), Ni(1)—O(1W), and Ni(1)—O(2) are 0.2003(2), 0.2050(3), 0.2036(2), 0.2067(2), 0.2070(2), and 0.2079(2) nm, respectively. And similar values have been observed in nickel complex of taurine salicylaldehyde Schiff base.<sup>5</sup> As expected all other bond distances and angles are in normal range and in good agreement with those of other taurine-containing complexes.<sup>3-7</sup> The complex **1** is a three-dimensional network with hydrogen bonds.

#### Structure of $[\text{Cu}(\text{TCSSB})(\text{H}_2\text{O})_2]_2 \cdot [\text{Cu}(\text{TCSSB})_2] \cdot 6\text{H}_2\text{O}$ (**2**)

In complex **2**, the coordination modes of the two copper ions are different. The Cu(1) is five-coordinated by two oxygen atoms [O(1) and O(2)] and one nitrogen atom from taurine condensation 5-chlorosalicylaldehyde Schiff base and two oxygen atoms from two water molecules with oxygen atom [O(1W)] of water at the apical position. So Cu(1) ion exhibits a very slightly distorted square pyramidal. The distance of Cu(1)—O(1W) [0.2413(3) nm] is longer than that of Cu(1)—O(2W) [0.1961(3) nm] because of O(1W) being at the apical position of square pyramidal. The geometry about Cu(2) is a four-coordinated distorted square-plane with

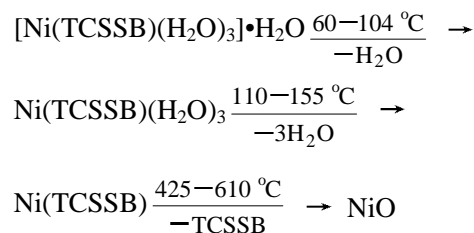
TCSSB. The total angle of O(5)-Cu(2)-N(2) ( $91.87^\circ$ ), O(5A)-Cu(2)-N(2) ( $88.13^\circ$ ), O(5)-Cu(2)-N(2A) ( $88.13^\circ$ ), O(5A)-Cu(2)-N(2A) ( $91.87^\circ$ ) is  $360^\circ$ . The atoms of O(5), O(5A), N(2), N(2A), Cu(2) belong to a plane. Cu(2) ion is a symmetric center of  $[\text{Cu}(\text{TCSSB})_2]$ . Two sulfo groups exist at the diagonal of the plane, forming anti-coordination molecule. The distance of S(1)—O(2) [0.1471(3) nm] is longer than the other S—O bond of complex **2** meaning that S(1)—O(2) bond has been stretched because O(2) is coordinated. The Cu(1)—N(1) distance is 0.1974 nm, which is slightly shorter than that of Cu(2)—N(2) (0.2018 nm). It is may be because two conjugation six-membered chelate rings have been formed by two oxygen and one nitrogen atoms from TCSSB. The O atoms of sulfo group between Cu-(TCSSB)<sub>2</sub> and Cu(TCSSB)(H<sub>2</sub>O)<sub>2</sub> are different because they have not been coordinated in Cu(TCSSB)<sub>2</sub>. The complex **2** is also a three-dimensional network complex of infinite length connected by hydrogen bonds.

The Cu(1)—O(1) (O of TCSSB) distance of the complex **2** is 0.1902 nm which is slightly longer than that of Cu(1)—O(1) (O of taurine salicylaldehyde Schiff base) (0.1882 nm) of the complex,<sup>6</sup> showing that the coordination ability of taurine condensation salicylaldehyde Schiff base is stronger than that of taurine condensation 5-chlorosalicylaldehyde Schiff base. It is may be because chlorine is an electro-attracting group, resulting in that electron density of oxygen in phenolic hydroxyl is reduced and the coordination capacity of this is abated. In those complexes, the M—O (O of sulfo group)

distances between 0.19 and 0.22 nm are also shorter than those of the M—O of the complexes<sup>10-12</sup> synthesized in water solvent. The structure of the complexes are different from that of [Cu(TSSB)H<sub>2</sub>O]<sub>2</sub>•2H<sub>2</sub>O,<sup>4</sup> [Cu(TSSB)(phen)]•1.5H<sub>2</sub>O<sup>6</sup> and [Ni(TSSB)(bpy)H<sub>2</sub>O]•4H<sub>2</sub>O<sup>5</sup> (TSSB = taurine salicylaldehyde Schiff base, phen = *o*-phenanthroline, bpy = 2,2'-bipyridine).

The TG-DTG curve was obtained using a TG209 thermal analyzer of NETZSCH. The heating rate was 10 °C•min<sup>-1</sup> and the flow rate of air was 20 mL•min<sup>-1</sup>. The TG-DTG thermogram of the complex **1** revealed that the compound decomposition takes place in three steps. The TG curve shows that the first stage mass loss is 4.67% between 60 and 104 °C, which coincides with the calculated value (4.59%) of losing 1 mol crystal water from the complex. In the second stage, Ni(TCSSB)(H<sub>2</sub>O)<sub>3</sub> is decomposed at 110—155 °C, with

the mass loss of 13.55%, the theoretical value being 13.76%, corresponding to the loss of 3 mol of coordination water. In the third stage, Ni(TCSSB) is decomposed at 425—610 °C, with the mass loss of 62.38%, the theoretical value being 62.60%, corresponding to the loss of 1 mol of TCSSB and the formation of NiO. From the above analysis, the thermal decomposition process of [Ni(TCSSB)(H<sub>2</sub>O)<sub>3</sub>]•H<sub>2</sub>O may be expressed by the following scheme:



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