DOI:10.14102/j.cnki.0254-5861.2005.06.004

Synthesis, Characterization and Structure of the Dimmer Complex $[Ni(TSSB)(phen)H_2O]\cdot C_2H_5OH\cdot 0.5H_2O$①

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ABSTRACT The title complex $[Ni(TSSB)(phen)H_2O]\cdot C_2H_5OH\cdot 0.5H_2O$ ($C_{23}H_{26}N_3NiO_{6.5}S$, TSSB = taurine salicylaldehyde Schiff base, phen = 1,10-phenanthroline) has been synthesized by the reaction of taurine salicylaldehyde Schiff base (TSSB), 1,10-phenanthroline and nickel acetate in water-ethanol. Its single-crystal structure was determined by X-ray diffraction method. The crystal belongs to triclinic, space group $P\bar{1}$ with a = 1.0562(2), b = 1.1604(2), c = 2.1170(3) nm, α = 103.257(3), β = 96.958(4), γ = 105.179(3)°, M_r = 539.24, V = 2.3917(6) nm^3, Z = 4, D_c = 1.498 g/cm^3, μ = 0.945 mm^{-1} and F(000) = 1124. The compound is a one-dimensional network, infinitely extending with hydrogen bonds. The Ni(Ⅱ) is 6-coordinated by one nitrogen and two oxygen atoms from taurine salicylaldehyde Schiff base, two nitrogen from 1,10-phenanthroline and one oxygen from water to form a distorted octahedronal coordination geometry.

Keywords: nickel complex, taurine-salicylaldehyde Schiff base, crystal structure, dimmer

1 INTRODUCTION

Schiff base complexes containing sulfur and complexes of amino acid Schiff bases[1~5] have recently aroused increasing interest because of their antiviral, anticancer and antibacterial activities. Taurine, an amino acid containing sulfur, is indispensable to human beings and has important physiological functions. Recently, we have found that the Shiff base derived from taurine has manifold coordination modes[4~10]. Aromatic-ring stacking interaction is an important characteristic of ternary complex and has important functions in organism such as stabilizing the double helical structure of DNA[11] and the interactions between anticancer drugs and DNA[12]. We report here in the synthesis and crystal structure of a new nickel(II) complex prepared by the reaction of $Ni(Ac)_2\cdot 4H_2O$ and sodium salt of Schiff base ligand 2-{[2′-hydroxyphenyl]methylidyne}nitriloethylsulforic acid derived from the reaction of taurine and Salicylaldehyde as well as 1,10-phenanthroline.

2 EXPERIMENTAL

Received 15 October 2004; accepted 17 December 2004 (**CCDC 273743**)

① Supported by the NSF of Guangxi Province (No. 0339034), the Science Research Foundation of Guangxi Universities and Minister of Education Foundation of Guangxi (No. [2004]20)

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2. 1 Materials and physical measurements

All solution and chemicals were commercial reagents and used without further purification. The elemental analysis was performed on a PE 1700 CHN auto elemental analyzer. The crystal structure was determined by a Bruker CCD Area Detector diffractometer and SHELXL crystallographic software of molecular structure.

2. 2 Synthesis

The complex was prepared by mixing an ethanol-water solution of salicylaldehyde (0.001 mol), taurine (0.001 mol) and sodium hydroxide (0.001 mol) with heating and stirring. After two hours, an aqueous solution containing 0.001 mol nickel acetate was added drop wise under stirring. The pH value of the mixture was adjusted to 5.5～6 with 0.5 mol/L HCl solution, followed by the drop wise addition of an ethanol solution containing 0.001 mol Phen with stirring. The resulting solution was left at room temperature, and blue crystals of the title complex were obtained after 15 days. Found (%): C, 51.26; H, 4.85; N, 7.82; S, 5.91. $C_{23}H_{26}N_3NiO_{6.5}S$ requires (%): C, 51.23; H, 4.86; N, 7.79; S 5.95.

2. 3 Crystal structure determination

A blue single crystal of the title compound with approximate dimensions of 0.40mm × 0.30mm × 0.10 mm was mounted on a glass fiber. X-ray diffraction intensity data were collected on a Bruker CCD Area Detector equipped with a graphite-monochromated Mo-$K\alpha$ radiation (λ = 0.071073 nm) by using an ω-2θ scan mode in the range of 1.89$\leqslant\theta\leqslant$27.60º at 294(2) K. A total of 16483 independent reflec- tions were measured, of which 10921 were unique (R_{int} = 0.0388) and 6466 were observed ($I > 2\sigma(I)$) and used in the subsequent structure determination and full-matrix least-square refinements. Absorption correction was performed by the SADABS program[13]. The structure was solved by direct methods and subsequent difference Fourier syntheses, revealing the positions of all non-hydrogen atoms. The hydrogen atoms were located geometrically. All non-hydrogen atoms were refined anisotropically. The final R = 0.0549 and R_w=0.1269 (w=1/[$\sigma^2(F_o^2)$+(0.0600P)^2], where P = (F_o^2 + 2F_c^2)/3) for 6466 observed reflec- tions with $I>2\sigma(I)$. S=1.061, $(\Delta/\sigma)_{max}$=0.003, $(\Delta\rho)_{max}$ = 848 and $(\Delta\rho)_{min}$ =–490 e/nm^3. The atomic coordinates and equivalent isotropic thermal parameters for the title complex are given in Table 1. The selected bond lengths and bond angles are listed in Table 2. All calculations were performed by using SHELXTL package[14].

Table 1. Atomic Coordinates (× 10^4) and Thermal Displacement Parameters (Å^2 × 10^3) of the Complex

Atom	*x*	*y*	*z*	U_{eq}	Atom	*x*	*y*	*z*	U_{eq}
Ni(1)	2460(1)	6828(1)	3136(1)	36(1)	Ni(2)	4187(1)	3517(1)	1697(1)	30(1)
S(1)	748(1)	8785(1)	3040(1)	47(1)	S(2)	5914(1)	1874(1)	2293(1)	39(1)
O(1W)	3003(2)	6704(2)	2198(1)	46(1)	O(2W)	4866(2)	4929(2)	2570(1)	43(1)
O(1)	2939(2)	5303(2)	3211(1)	41(1)	O(5)	2780(2)	4291(2)	1448(1)	38(1)
O(2)	1987(2)	8435(2)	3045(1)	52(1)	O(6)	5805(2)	2816(2)	1953(1)	43(1)
O(3)	696(3)	9570(2)	3654(1)	73(1)	O(7)	6609(2)	1064(2)	1970(1)	60(1)
O(4)	520(2)	9278(2)	2475(1)	62(1)	O(8)	6470(2)	2421(2)	2991(1)	61(1)
N(1)	522(2)	5774(2)	2743(1)	36(1)	N(4)	2892(2)	2344(2)	2077(1)	33(1)
N(2)	4420(3)	7908(2)	3592(1)	47(1)	N(5)	3843(2)	2234(2)	768(1)	37(1)
N(3)	2296(3)	7161(2)	4135(1)	43(1)	N(6)	5471(2)	4561(2)	1210(1)	38(1)
C(1)	2109(3)	4378(3)	3355(1)	37(1)	C(22)	1495(3)	3759(3)	1340(1)	34(1)
C(2)	2624(4)	3681(3)	3722(2)	50(1)	C(23)	618(3)	4250(3)	1013(2)	45(1)
C(3)	1797(4)	2650(3)	3846(2)	59(1)	C(24)	–748(3)	3782(3)	917(2)	50(1)
C(4)	446(4)	2274(3)	3620(2)	57(1)	C(25)	–1322(3)	2784(3)	1149(2)	52(1)
C(5)	–87(3)	2957(3)	3282(2)	48(1)	C(26)	–513(3)	2270(3)	1462(2)	44(1)
C(6)	715(3)	4027(3)	3150(1)	37(1)	C(27)	901(3)	2720(3)	1568(1)	35(1)
C(7)	31(3)	4694(3)	2806(1)	38(1)	C(28)	1629(3)	2156(3)	1962(1)	35(1)
C(8)	–410(3)	6285(3)	2407(2)	46(1)	C(29)	3388(3)	1692(3)	2532(2)	44(1)
C(9)	–599(3)	7397(3)	2888(2)	50(1)	C(30)	4275(3)	948(3)	2247(2)	43(1)
C(10)	5443(4)	8282(4)	3313(2)	65(1)	C(31)	3033(4)	1092(3)	556(2)	53(1)

C(11)	6735(4)	8945(4)	3696(3)	85(2)	C(32)	2973(4)	296(4)	–57(2)	70(1)
C(12)	6922(4)	9173(4)	4368(3)	88(2)	C(33)	3787(4)	693(4)	–456(2)	68(1)
C(13)	5860(4)	8791(4)	4680(2)	65(1)	C(34)	4662(3)	1908(4)	–259(2)	53(1)
C(14)	5959(5)	9009(4)	5382(2)	82(2)	C(35)	5499(4)	2437(4)	–660(2)	68(1)
C(15)	4915(5)	8617(4)	5641(2)	81(1)	C(36)	6239(3)	3631(4)	–457(2)	62(1)
C(16)	3617(4)	7967(3)	5239(2)	61(1)	C(37)	6276(3)	4397(3)	175(2)	50(1)
C(17)	2490(5)	7573(4)	5491(2)	75(1)	C(38)	7026(3)	5645(4)	417(2)	59(1)
C(18)	1285(5)	6968(4)	5074(2)	75(1)	C(39)	6988(3)	6313(3)	1029(2)	59(1)
C(19)	1235(4)	6774(3)	4391(2)	54(1)	C(40)	6199(3)	5729(3)	1421(2)	48(1)
C(20)	3486(3)	7751(3)	4548(2)	46(1)	C(41)	5502(3)	3896(3)	598(1)	37(1)
C(21)	4621(3)	8156(3)	4266(2)	50(1)	C(42)	4658(3)	2657(3)	370(1)	36(1)
O(9)	7200(3)	5026(3)	3407(1)	93(1)	C(45)	857(5)	7749(5)	861(2)	102(2)
C(43)	7426(6)	5535(7)	4094(3)	136(2)	C(46)	78(4)	8520(4)	702(2)	86(2)
C(44)	6292(7)	5418(8)	4351(3)	169(3)	O(3W)	7741(3)	–851(3)	2136(2)	112(1)
O(10)	1752(3)	8134(3)	1465(1)	85(1)					

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Table 2. Selected Bond Lengths (Å) and Bond Angles (°)

Bond	Dist	Bond	Dist	Bond	Dist
Ni(1)–O(1)	1.998(2)	Ni(1)–N(3)	2.098(3)	Ni(2)–O(2W)	2.0712(2)
Ni(1)–N(1)	2.046(2)	Ni(1)−O(1W)	2.116(2)	Ni(2)–N(5)	2.101(2)
Ni(1)–N(2)	2.095(3)	Ni(2)–O(5)	2.005(2)	Ni(2)–N(6)	2.102(2)
Ni(1)–O(2)	2.096(2)	Ni(2)–N(4)	2.040(2)	Ni(2)–O(6)	2.140(2)
Angle	(°)	Angle	(°)	Angle	(°)
O(1)–Ni(1)–N(1)	89.88(9)	O(1)–Ni(1)–O(1W)	92.35(9)	O(2W)–Ni(2)–N(5)	169.33(9)
O(1)–Ni(1)–N(2)	89.70(10)	N(1)–Ni(1)–O(1W)	93.43(9)	O(5)–Ni(2)–N(6)	89.59(9)
N(1)–Ni(1)–N(2)	176.75(10)	N(2)–Ni(1)–O(1W)	89.81(10)	N(4)–Ni(2)–N(6)	173.62(9)
O(1)–Ni(1)–O(2)	178.82(9)	O(2)–Ni(1)–O(1W)	86.62(9)	O(2W)–Ni(2)–N(6)	91.28(9)
N(1)–Ni(1)–O(2)	90.75(9)	N(3)–Ni(1)–O(1W)	169.17(9)	N(5)–Ni(2)–N(6)	78.97(9)
N(2)–Ni(1)–O(2)	89.73(10)	O(5)–Ni(2)–N(4)	91.31(9)	O(5)–Ni(2)–O(6)	175.42(8)
O(1)–Ni(1)–N(3)	87.95(9)	O(5)–Ni(2)–O(2W)	88.69(8)	N(4)–Ni(2)–O(6)	93.10(9)
N(1)–Ni(1)–N(3)	97.40(10)	N(4)–Ni(2)–O(2W)	95.05(9)	O(2W)–Ni(2)–O(6)	89.69(8)
N(2)–Ni(1)–N(3)	79.36(11)	O(5)–Ni(2)–N(5)	95.50(9)	N(5)–Ni(2)–O(6)	85.38(9)
O(2)–Ni(1)–N(3)	92.95(10)	N(4)–Ni(2)–N(5)	94.66(9)	N(6)–Ni(2)–O(6)	86.16(9)

Table 3. Hydrogen Bond Lengths (10^{-1} nm) and Bond Angles (°)

D–H–A	d(D–H)	d(H–A)	d(D–A)	∠DHA
O(1W)–H(1WA)···O(10)	0.86	2.10	2.957(4)	173.1
O(1W)–H(1WB)···O(5)	0.86	1.97	2.818(3)	166.6
O(2W)–H(2WA)···O(1)	0.86	1.82	2.652(3)	163.4
O(2W)–H(2WB)···O(9)	0.86	1.98	2.816(4)	164.0
O(9)–H(9)···O(8)	0.82	2.00	2.811(4)	168.0
O(10)–H(10A)···O(4)	0.82	2.06	2.882(4)	177.4
O(3W)–H(3WA)···O(7)	0.86	2.00	2.854(4)	174.0

3 RESULTS AND DISCUSSION

As shown in Fig. 1, the molecule of the title compound is a dimmer. The nickel (II) cations are coordinated by one N and two O atoms (O(1) and O(2)) from taurine salicylaldehyde Sciff base, two N atoms from 1,10-phenanthroline and one O atom from water molecule forming a slightly distorted octahedron geometry with O(1) and O(2) at the apical positions. The sum of bond angles N(1)–Ni(1)–N(3) (97.40º), N(2)–Ni(1)–N(3) (79.36º), N(1)–Ni(1)–O(1W) (93.43º) and N(2)–Ni(1)–O(1W) (89.81º) is 360.00º, showing the N(1), N(3), N(2) and O(1W) atoms are coplanar. The interatomic distance of Ni(1)–O(1) is 0.1998(2) nm, Ni(1)–N(1) 0.2046(2) nm, Ni(1)–N(2) 0.2095(3) nm, Ni(1)–O(2) 0.2096(2) nm, Ni(1)–N(3)

0.2098(3) nm and Ni(1)–O(1W) 0.2116(2) nm. Similar values have been observed in nickel complex of salicylaldehyde Schiff base. As expected, all other bond distances and bond angles are within the normal ranges, in good agreement with those of other taurine-containing complexes[4～6, 8～10]. The bond length of Ni(1)–O(1) (0.1998(2) nm) is shorter than that of Ni(1)–N(1) (0.2046(2) nm), indicating the oxygen atom of hydroxyl has stronger coordination ability than the nitrogen atom of imine. Coordination of Ni(2) is similar to that of Ni(1), but the corresponding bond lengths are slightly different (Ni(2)–O(5) 0.2005(2) nm, Ni(2)–N(4) 0.2040(2) nm, Ni(2)–O(2W) 0.2071(2) nm, Ni(2)–N(5) 0.2101(2) nm, Ni(2)–N(6) 0.2102(2) nm and Ni(2)–O(6) 0.2140(2) nm).

There exist a number of intra- and intermolecular hydrogen bonds in the title compound (Fig. 2). The complex is linked by hydrogen bonds between O(1) and O(2W) as well as O(5) and O(1W) together with other hydrogen bonds listed in Table 3 to form a one-dimensional chain structure.

In the compound, coordination of Schiff base is different from that discussed in references [6～8]. In references [6] and [8], Schiff bases are bidentate ligands (both O atom of phenolic hydroxyl and N atom of imidogen take part in coordination); while in reference [7], it is the O atoms of sulfonic group instead of O atom of phenolic hydroxyl and N atom of imidogen that are coordinated. In the compound, coordination of Schiff base (TSSB) is the same as that in references [4] and [5] because TSSB is a tridentate ligand.

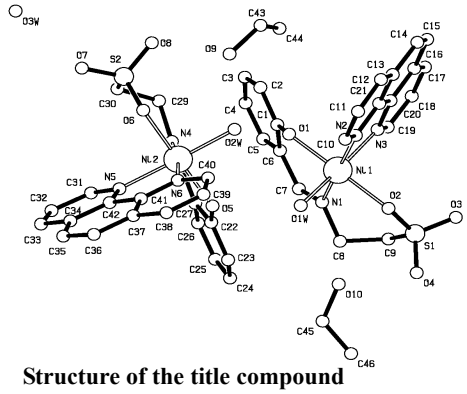

Fig. 1. Structure of the title compound

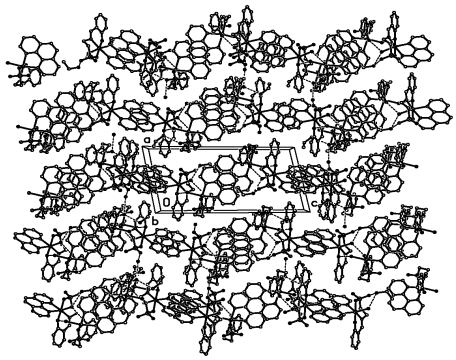

Fig. 2. Packing diagram of the title compound

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Synthesis and Crystal Structure of a Heterotrinuclear Compound $[PdCu_2(tdt)(\mu\text{-dppm})_2(MeCN)_4](ClO_4)_2\cdot 2/3H_2O$ (tdt=3,4-Toluenedithiolate, dppm=Bis(diphenylphosphino)methane)

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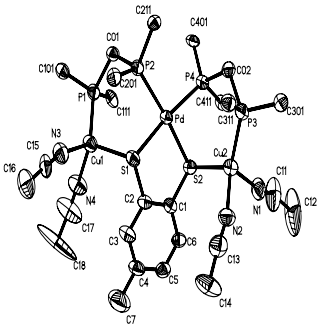
CHEN Zhong-Ning

Chinese J. Struct. Chem., 24, 621(2005)



The title compound **1** has been prepared and characterized by X-ray crystallography. The dithiolate tdt (tdt = 3, 4-toluenedithiolate) displays a chelating and bridging coordination mode, which chelates a Pd^{II} atom and bridges two Cu^{I} atoms. The Pd^{II} center is located in an approximate square-planar environment with S_2P_2 donors, and each Cu^{I} center exhibits a tetrahedral geometry.

Synthesis and Crystal Structure of a Novel Complex $[Ag(NIT3Py)_3]^{\bullet}(ClO_4)(H_2O)$

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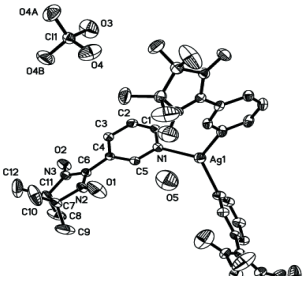
LIAO Dai-Zheng
JIANG Zong-Hui
YAN Shi-Ping

Chinese J. Struct. Chem., 24, 625(2005)

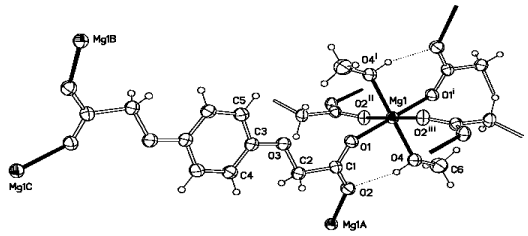


The title complex has been synthesized and structurally characterized by X-ray diffraction methods. The structure consists of $Ag(NIT3Py)_3^{+}$ moiety, one water molecule and one perchloric ion. The Ag(I) ion is in a trigonal planar environment formed by three nitrogen atoms from three NIT3Py.

Synthesis and Crystal Structure of a Novel 3D Magnesium (II) Coordination Polymer Constructed by Flexible Benzene-1,4-dioxyacetate

GAO Shan HUO Li-Hua
CHENG Xiao-Li
LIU Ji-Wei

Chinese J. Struct. Chem., 24, 629(2005)



A novel magnesium(II) coordination polymer $[Mg(1,4\text{-}BDOA)(CH_3OH)_2]_n$ (1,4-$BDOA^{2-}$ = benzene-1,4-dioxyacetate dianion) has been synthesized and characterized. The Mg(II) atoms adopting an octahedral geometry are linked by 1,4-$BDOA^{2-}$ ligands into a 3D infinite network structure.

Synthesis, Characterization and Structure of the Dimmer Complex $[Ni(TSSB)(phen)\text{-}H_2O]\cdot C_2H_5OH\cdot 0.5H_2O$

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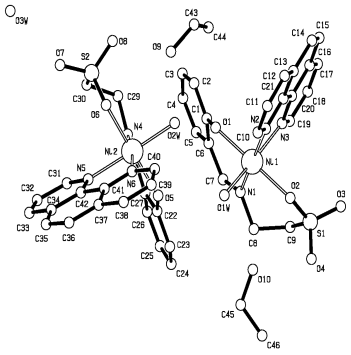
Chinese J. Struct. Chem., 24, 633(2005)



The title complex has been synthesized and its crystal structure was determined. The compound is a 1D network infinitely extending with hydrogen bonds. The Ni(II) is coordinated by one nitrogen and two oxygen atoms from taurine salicylaldehyde Schiff base, two nitrogen from 1,10-phenanthroline and one oxygen from water to form a distorted octahedron coordination geometry.