

Synthesis, Crystal Structure, and Properties of the Enantiotopic Complex Constructed from Chiral Ligand H₂bpb, [Co (H₂bpb)₂ (NCS)₂ (CH₃OH)₂] (H₂bpb = 1, 2-Bis (3-Pyridylcarboxamide)Benzene)

Ping Zhang, Ben-Lai Wu, Yun-Yin Niu, Hong-Yun Zhang, Cao-Yuan Niu, and Hong-Wei Hou

Department of Chemistry, Zhengzhou University, Zhengzhou, P. R. China

A six-coordinating cobalt(II) complex, $[Co(H_2bpb)_2(NCS)_2(CH_3OH)_2]$, where H_2bpb is 1, 2-bis(3-pyridylcarboxamide) benzene, was prepared and characterized by IR spectrum and thermal analysis, and its crystal structure was determined X-ray analysis. It crystallizes in the triclinic system with space group P-1 with a =8.7005(9) Å, b = 9.6114(10) Å, c = 13.767(2) Å, $\alpha = 101.393(2)$, $\beta =$ 93.185(2), $\gamma = 114.5180(10)$, V = 1014.5(2) Å, Z = 1. Fluorescent and thermal properties of the complex were also investigated.

Keywords cobalt(II) complex, crystal structure, fluorescent, thermal analysis

INTRODUCTION

The syntheses of ligands based on 1,2-disubstituted benzene have attracted a great deal of attention, with a broad array of both acyclic and macrocyclic ligands reported.^[1] In particular,diamides incorporating a –CO–NH–benzene–NH–CO– core based on 1,2-benzenediamine have been examined, with interest in these compounds extending to potential applications as diverse as asymmetric catalysis,^[2] molecular receptors,^[3] dendrite synthesis,^[4] and platinum(II) complexes with antitumor properties.^[5] The carboxamide [–C(O)NH–] group, which is the primary structure of proteins, is all over in the nature and an important ligand construction unit for coordination chemists.^[6] In the control of molecular architectures pyridine carboxamide ligands are used extensively.^[7]

Coordination compounds containing the thiocyanate ligand are usually reported.^[8] Thiocyanate plays a diverse role in the

structural chemistry and the kinetics of reactions.^[9] Cobalt complexes are useful in highly selective catalytic oxidation reactions using molecular oxygen, as models for oxygenases, peroxydases, or mono- and dioxygenases , for example, catalyzing the oxidation of phenols, alcohols, flavonoides, nitroalkanes, hydrazines, or olefins. While extended coordination networks derived from pyridylamide ligands have been reported with the metal centers Ag(I), Au(I), Pd(II), and Pt(II),^[10–12] compounds similar to Co(II) have not been studied extensively. In the present communication, we have reported the synthesis of the complex [Co (H₂bpb)₂(NCS)₂(CH₃OH)₂] (H₂bpb = 1, 2-bis(3-pyridylcarboxamide)benzene) and turned our attention to characterization of the complex.

EXPERIMENTAL

Materials and Methods

Nicotinic acid was analytical pure grade from Alfa Aesar, phthalamine and other reagents were also analytical pure grade and purchased from TianJin Reagent Factory, used without further purification. The 1, 2-bis(3-pyridylcarboxamide)benzene (H₂bpb) was synthesized following a procedure reported previously^[13] with a yield of 70%. Thionyl chloride and pyridine were redistilled. Melting points were taken on an XT-5 microscope melting point apparatus. IR spectra were recorded on a Nicolet IR-470 spectrometer from KBr pellets in range 4000–400 cm⁻¹. Crystallographic data were measured on a Bruker APEX-II area-detector diffractometer with Mo-Ka radiation ($\lambda = 0.71073$ Å). Thermal analysis curve was scanned in the range of 35–800°C with argon atmosphere on STA 409 PC thermal analyzer. The fluorescence spectra were recorded in the solid state at room temperature on HITRCHI F-4500 Fluorophotometer.

Synthesis of the Complex [Co(H₂bpb)₂(NCS)₂(CH₃OH)₂]

At room temperature, a clear DMF solution (5 mL) of H_2 bpb (63.7 mg, 0.2 mmol) was slowly added to a methanol solution (10 mL) of CoSO₄ (56.2 mg, 0.2 mmol), then 5 mL solution

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Address correspondence to Hong-Yun Zhang, Department of Chemistry, Zhengshou University, Zhengzhou 450052, P. R. China. E-mail: wzhy917@zzu.edu.cn

TABLE 1 Crystallographic data and structure refinement for complex

Structural parameter			
Empirical formula	C40 H36 Co N10 O6 S2		
M	875.84		
Crystal group	Triclinic		
Space group	P-1		
a/Å	8.7005(9)		
b/Å	9.6114(10)		
c/Å	13.767(2)		
$\alpha /^{\circ}$	101.393(2)		
βI°	93.185(2)		
γI°	114.5180(10)		
V/Å ³	1014.5(2)		
$D_c/g.cm^{-3}$	1.434		
Ζ	1		
μ/mm^{-1}	0.586		
Crystal size/mm ³	$0.34 \times 0.24 \times 0.23$		
Refins collected/Unique	7811/3769		
$\frac{R1, \omega R2[I > 2\sigma(I)]}{2\sigma(I)}$	$0.0298^a; 0.0802^b$		

 ${}^{a}\mathbf{R}_{1} = \Sigma ||F_{o}| - |F_{c}|| / \Sigma |F_{o}|.$ ${}^{b}\omega\mathbf{R}_{2} = [\Sigma (||F_{o}| - |F_{c}||)^{2} / \Sigma \omega |F_{o}|^{2}]^{1/2}.$

of potassium thiocyanate (19.4 mg, 0.2 mmol) in methanol was added to the above mixture with continuous stirring for 30 min and filtered. The filtrate was allowed undisturbed evaporation at ambient temperature, and pink block crystals suitable for X-ray single crystal diffraction analysis were collected by being filtered and washed with methanol and acetone for one week. Yield: 40%. IR (KBr, $v \text{ cm}^{-1}$): 3319 m, 3271 m, 2052 s, 1657 s, 1598 s, 1533 s, 1317 s, 1199 m, 1028 m, 918 m, 771 m, 702 m.

X-Ray Structure Determination

Crystallographic data for the title compound were collected at 291(2) K on a Bruker APEX-II area-detector diffractometer with Mo-Ka radiation ($\lambda = 0.71073$ Å). Absorption corrections were applied by using SADABS. The structure was solved with direct methods and refined with full-matrix least-squares techniques on F^2 using the SHELXTL program package.^[14] All of the non-hydrogen atoms were refined anisotropically. The hydrogen atoms were assigned with common isotropic displacement factors and included in the final refinement by using geometrical restraints. Crystal data are summarized in detail in Table 1. Selected bond lengths and angles are shown in Table 2.

RESULTS AND DISCUSSION

IR Spectra

In IR spectrum of the free ligand H_2 bpb, a band of medium intensity, shows at 3282 cm⁻¹ and is recognized as the ν (NH).^[15]

TABLE 2 Selected bond distances (Å) and angles (°) for cobalt(α) complex

eompies	n
Co(1)-N(5)	2.0814(16)
Co(1)-N(5)#1	2.0814(16)
Co(1)-O(3)	2.1011(14)
Co(1)-O(3)#1	2.1011(14)
Co(1)-N(1)	2.1855(14)
Co(1)-N(1)#1	2.1855(14)
N(5)-Co(1)-N(5)#1	180.0
N(5)-Co(1)-O(3)	88.50(7)
N(5)#1-Co(1)-O(3)	91.50(7)
N(5)-Co(1)-O(3)#1	91.50(7)
N(5)#1-Co(1)-O(3)#1	88.50(7)
O(3)-Co(1)-O(3)#1	179.999(1)
N(5)-Co(1)-N(1)	90.16(6)
N(5)#1-Co(1)-N(1)	89.84(6)
O(3)-Co(1)-N(1)	89.73(6)
O(3)#1-Co(1)-N(1)	90.27(6)
N(5)-Co(1)-N(1)#1	89.84(6)
N(5)#1-Co(1)-N(1)#1	90.16(6)
O(3)-Co(1)-N(1)#1	90.27(6)
O(3)#1-Co(1)-N(1)#1	89.73(6)
N(1)-Co(1)-N(1)#1	180.0

Symmetry transformations used to generate equivalent atoms: #1 -x + 2, -y + 1, -z + 1.

The characteristic absorption bands at 1665, 1529, and 1283 cm⁻¹ for H₂bpb are due to amide \Box [mainly ν (C=O)], amide II [mainly ν (C=N)], and amide III [mainly δ (N-H)] vibrations, respectively.

The complex has characteristic IR bands at 1657, 1533, and 1316 cm^{-1} .^[16] The amide \Box [mainly ν (C=O)] did not disappear and trivially shift to 1657 cm⁻¹, which suggests that in the complex H₂bpb still is in keto-form and the non-coordination of this fragment. Comparing the corresponding bands of the complex with the free ligand, the obvious changes are found in amide \Box and III vibrations. The amide I band shifts to a lower frequency and the amide III band shifts to a higher frequency. These facts may be explained by inter- and/or intramolecular hydrogen bonding and C=O group and N-H group did not

TABLE 3 Hydrogen bonds for L (Å and $^{\circ}$)

D-HA $d(D-H)$ $d(HA)$ $d(DA)$ $<(DHA)$ N(2)-H(2N)O(2)#20.862.072.8979(18)161.7N(3)-H(3N)O(1)#30.862.132.8789(19)144.9O(3)-H(3E)N(4)#40.79(3)1.99(3)2.762(2)1.70(3)					
$\begin{array}{llllllllllllllllllllllllllllllllllll$	D-НА	d(D-H)	d(HA)	d(DA)	<(DHA)
	N(2)-H(2N)O(2)#2 N(3)-H(3N)O(1)#3 O(3)-H(3E)N(4)#4	0.86 0.86 0.79(3)	2.07 2.13 1.99(3)	2.8979(18) 2.8789(19) 2.762(2)	161.7 144.9 170(3)

Symmetry transformations used to generate equivalent atoms: #1 - x + 2, -y + 1, -z + 1, #2 - x + 1, -y + 1, -z, #3 - x + 1, -y, -z, #4 x + 1, y, z.



 $FIG. \ 1. \ Structure \ of \ [Co(H_2 bpb)_2 (NCS)_2 (CH_3 OH)_2] \ with \ the \ atom \ labeling \ scheme \ (30\% \ probability \ ellipsoids). \ Hydrogen \ atoms \ have \ been \ omitted \ for \ clarity.$



FIG. 2. Two chiral conformations of H_2 bpb (hydrogen atoms have been omitted for clarity).



FIG. 3. One-dimensional ordering and of hydrogen-bond system [Co (H2bpb)2(NCS)2(CH3OH)2].



FIG. 4. Structure of the dimeric unit of $[Co (H_2bpb)_2 (NCS)_2 (CH_3OH)_2]$ with the atom labeling scheme (parts of hydrogen atoms have been omitted for clarity).

take part in coordination with metal. The IR spectrum of the complex exhibits additional strong absorption bands at 2052 cm^{-1} , which should be ascribed to the occurrence of the SCN group.

Described Crystal Structure of the Complex

The crystal structure of the complex, $[Co (H_2bpb)_2(NCS)_2]$ (CH₃OH)₂], is shown in Figure 1 and selected bond distances and angles are listed in Table 2. The cobalt ion sits on a crystallographically imposed center. Two H₂bpb ligands as monodentate occupy two equatorial positions in trans through one pyridine nitrogen coordinating to Co (II) respectively. There are two thiocyanates on the position of equatorial plane through nitrogen atom also coordinating to Co(II) in trans respectively. Axial positions are occupied by two oxygen atoms of two methanol molecules. Thus, the cobalt ion has an octahedral geometry, comprising CoN₄O₂ architectural coordination environment. The ligand bite angles of N1(py)-CoII-O3(methanol) are 89.73(6)° and 90.27(6)°, respectively; those of N5(thiocyanate)-CoII-O3(methanol) are 88.50(7)° and 91.50(7)°, respectively; those of N1(py)-CoII-N5 (thiocyanate) are $90.16(6)^{\circ}$ and $89.84(6)^{\circ}$, respectively (Table 2). The bond angles around $Co(\alpha)$ are fairly close to 90°, indicating a slight distortion of the octahedral coordination sphere. Mean value (2.1855(14) Å) of Co-N (py) lengths is appreciably longer than literature mean value (2.117(3) Å).^[16] In this conformation, plane I [C(7), C(8), C(9), C(10), C(11), and C(12)] makes dihedral angles 91.8° and 145.1° with plane II [N(1), C(1), C(2), C(3), C(4), and C(5)] and plane III [N(4), C(14), C(15), C(16), C(17), and C(18)], respectively. Plane II makes a dihedral angle



FIG. 5. The stereo drawings of the crystal packing of the complex viewed along b axis.



FIG. 6. TG/DSC curves for complex $[Co (H_2bpb)_2(NCS)_2(CH_3OH)_2]$. The curves were obtained in dynamic nitrogen atmosphere (approximately 50.0 mL min⁻¹) and heating rate of $10^{\circ}C \text{ min}^{-1}$.

 62.0° with the plane III (as in Figure 1). These dihedral angles reveal that all three aromatic rings are not coplanar to each other in crystal structural unit.

Due to stereo-isomer influencing, the ligand H_2 bpb, which coordinates cobalt(II) in trans form through one pyridine nitrogen atom, exists in the chiral conformations A or B in solid state the complex, as shown in Figure 2. It is seen from Figure 1 that two H_2 bpb and two thiocyanates coordinate to Co(II) in trans form, respectively, to form the complex having reverse center of symmetry and reverse plane of symmetry. So the chiral ligand H_2 bpb coordinates to Co(II) to form a mononucleated enantiotopic complex.

The pyridine nitrogens N(3) and methanol hydroxyls form strong intermolecular hydrogen bonds (O(3)-H(3E)···N(4) = 2.762(2) Å). The molecules of the complex are linked together through solvent-mediated hydrogen bonding of the kind $Co(\alpha)$ ···OH(Me)···N(Py) and form into 1D chain by slantly alternating bc plane (Figure 3). In 1D chains H₂bpb ligands do not parallel each other, which has a natural helicity. The solvate molecules of methanol play an important linkage part in the selfassembly process. Each ligand has four available NH and C=O groups available for intermolecular hydrogen bonding. Those 1D chains then self-assemble through intermolecular hydrogen bonds between carbonyl and amide groups (N(3)-H(3N)···O(1) = 2.8789(19) Å) of adjacent molecules (Figure 4). Thus, in irregular direction an intricate three-dimensional network comes into being,^[17] as shown in Figure 5.

Thermogravimetric Analysis (TG)

The thermal behavior of the complex under investigation was studied using the thermogravimetry (TG) and the DSC techniques. The DSC-TG curve of the complex (shown as in Figure 6) was scanned in the range of 30–800°C with argon atmosphere on STA 409 PC Thermal Analyzer. The thermal analytic data for initial complex show a first endothermic weight loss of 7.09% between 157 and 187°C with a DSC peak at 179°C which corresponds exactly (Calcd. 7.32%) to the release of two



FIG. 7. The fluorescence excitation spectra of ligand and the complex in solid state.

molecules of coordinated methanol of the complex. After loss of coordinated methanol, the TGA curve shows that the complex is stable up to 272°C. The intermediate [Co (H₂bpb)₂ (NCS)₂] is not thermally stable, because a new endothermic decomposition starts at 272°C. The degradation of the ligand H₂bpb is a complicated process and takes place in the temperature range 272–800°C. The thermal decomposition process of the H₂bpb is not easy to understand because of unclear decomposition steps. The mass loss observed between 270 and 351°C with a DSC peak at 272°C $\Delta m = 55.14\%$. At higher temperatures (351–800°C, a gradual mass loss totals 12.48%, corresponding to the elimination of two molecule of SCN (calculated value = 13.25%). The total experimental mass loss value is 75.04% and the final residue at 800°C may be the buildup of carbon and inorganic compound CoO.

Photoluminescent Properties

The fluorescence spectra of the ligand H₂bpb and the complex are determined in the solid state under the same measurement environment at room temperature on HITRCHI F-4500 Fluorophotometer. The fluorescence excitation spectra of the ligand H₂bpb ($\lambda_{max} = 244$ nm) and that of the complex ($\lambda_{max} =$



FIG. 8. The fluorescence emission spectra of ligand and the complex in solid state.

245 nm) are illustrated in Figure 7. The emission spectra of the ligand and the complex upon excitation at the isosbestic point at 245 nm are plotted in Figure 8. Though the complex displays an evidently higher luminescence strength than ligand H₂bpb ($\lambda_{max} = 389$ nm), but models and positions of their peaks are same, which is likely attributed to the charge transfer of intraligand.

SUPPLEMENTARY MATERIAL

Crystallographic data for the structural analyses have been deposited with the Cambridge Crystallographic Data Center, CCDC Nos. 609946, for the complex. Copies of this information may be obtained free of charge from: The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: +44 1223 336033; E-mail: deposit@ccdc.cam.ac.uk or www:hppt://www.ccdc.cam.ac.uk).

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