

Notes

New Supramolecular Complex Assembled through Hydrogen Bonds. Crystal Structure of $\text{Co}(\text{PMBP-tsc})_2 \cdot 2\text{DMF} \cdot 2\text{H}_2\text{O}$

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The structure of the complex $[\text{Co}(\text{PMBP-tsc})_2 \cdot 2\text{DMF} \cdot 2\text{H}_2\text{O}]$ (PMBP-tsc = 1-phenyl-3-methyl-4-benzoylpyrazol-5-one-thiosemicarbazone) has been determined by X-ray crystallography. It crystallizes in the orthorhombic system, space group $Pna2_1$, with lattice parameters $a = 2.1170(3)$ nm, $b = 1.2780(10)$ nm, $c = 1.8956(2)$ nm, $V = 4.5258(9)$ nm³ and $Z = 4$. The structure shows that Co^{2+} in the complex is hexacoordinated with a distorted octahedral coordination sphere. The water molecules bridge the adjacent stacks through hydrogen bonds and lead to supramolecular formation with three-dimensional network structure.

Keywords cobalt(II) compound, crystal structure, supramolecule, hydrogen bonds

Introduction

Molecular polymers with one dimensional or multi-dimensional structure assembled through hydrogen bonds is an important research content in the supra-molecular chemistry and crystal engineering.^{1,2} With the development of new type functional materials such as molecular magnetic, selected catalysis, reversible catalysis, reversible host-guest molecular (ion) exchange *etc.*,³ the molecular design and synthesis have already attracted considerable attention in supramolecular system. The supramolecular complexes and organic metal compounds containing hydrogen bond have also been extensively studied.⁴⁻⁶

In recent years investigations based on thiosemicar-

bazones and their metal complexes⁷⁻¹⁰ have been increased considerably because of their potentially biological activities such as antibacterial, antimalarial, antiviral, antineoplastic *etc.* Though 1-phenyl-3-methyl-4-benzoylpyrazol-5-one (HPMBP) has been widely studied as extraction and chelating agents of metal ions,¹¹⁻¹⁴ few work have been reported on the metal complexes.¹⁵ In this paper, the synthesis of the complex $\text{Co}(\text{PMBP-tsc})_2$ and the crystal structure of the supramolecular complex $[\text{Co}(\text{PMBP-tsc})_2 \cdot 2\text{DMF} \cdot 2\text{H}_2\text{O}]$ were reported. The water molecules bridge the molecules of the adjacent stacks through hydrogen bonds and lead to the formation of supramolecules with three-dimensional network structures.

Experimental

Materials

All solvents and chemicals were of commercial reagents and were used without further purification.

Physical measurements

The elemental analysis was performed on a PE 1700 CHN auto elemental analyzer. IR spectra were recorded on a Bio-RAD FTS-40 spectrometer as KBr disks. The crystal structure was determined by a Siemens P4 diffractometer and SHELXTL crystallographic software package

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of molecular structure.

Synthesis of the ligand

PMBP-tsc was prepared by mixing equimolar amounts (10 mmol) of 1-phenyl-3-methyl-4-benzoylpyrazol-5-one (HPMBP) and thiosemicarbazone in 100 mL of methanol containing a few drop of glacial acetic acid. The mixture was refluxed with an oil bath at 80 °C for 3 h and then cooled in the dark. Finally the white product was filtered, washed with ethanol, and dried *in vacuo* (yields ca. 80%). IR ν : 3470 (m, NH₂), 3341 [m, N(2)—H], 2537 (br, OH),¹⁵ 1577 (s, C=N), 1475 (s, pyrazolone-ring), 836 (m, C=S) cm⁻¹; Anal. calcd for C₁₈H₁₇N₅OS: C 61.52, H 4.87, N 19.93; found C 61.44, H 4.67, N 20.21.

Preparation of the complex Co(PMBP-tsc)₂

Co(OAC)₂·4H₂O (2 mmol) in water (20 mL) was added dropwise to a warm solution of PMBP-tsc (2 mmol) in anhydrous EtOH (60 mL). The mixture was then refluxed for 0.5 h with stirring, and the black product formed was filtered, washed with ethanol and dried *in vacuo*. IR ν : 3472 (w, NH₂), 1593 (s, C=O & S—C=N), 1563 (s, C=N), 1483 (s, pyrazolone-ring), 760 (m, C—S) cm⁻¹; Anal. calcd for Co(C₁₈-

H₁₆N₅OS)₂: C 56.91, H 4.25, N 18.44; found C 56.57, H 4.03, N 18.22.

Black single crystals suitable for X-ray diffraction analysis were obtained by slow evaporation of its DMF/C₂H₅OH solutions.

X-Ray structure determination

Cell constants and orientation matrix for data collection were obtained with least-squares refinement of 30 reflections in the range 2.85° < θ < 15.8° and 5845 reflections in range of 1.92° < θ < 28.00° were collected in ω -scan on a siemens P4 four-circle diffractometer using graphite-monochromated Mo K α (λ = 0.071073 nm). The structure was solved by direct methods. All non-hydrogen atoms were refined anisotropically. The hydrogen atoms of O(5) were located from the difference fourier map and others except for those of O(6) were located in their calculated positions. A summary of crystal data, experimental details and refinement results are listed in Table 1. Non-hydrogen atomic coordinates and equivalent isotropic displacement parameters are shown in Table 2. Selected bond lengths and bond angles are listed in Table 3. The molecular structure and packing diagram of the complex are shown in Fig. 1 and Fig. 2, respectively.

Table 1 Crystal data and details of the structure determination for Co(PMBP-tsc)₂·2DMF·2H₂O

Empirical formula	C ₄₂ H ₅₀ CoN ₁₂ O ₆ S ₂	Empirical formula	C ₄₂ H ₅₀ CoN ₁₂ O ₆ S ₂
Formula weight	941.99	θ range for data collection	1.92 to 28.00°
Temperature	296(2) K	Limiting indices	1 ≤ h ≤ 27, 0 ≤ k ≤ 14, 0 ≤ l ≤ 25
Wavelength	0.071073 nm	Reflections collected	6418
Crystal system	Orthorhombic	Independent reflections	5618 (R_{int} = 0.0110)
Space group	<i>Pna</i> 2 ₁	Max. and min. transmission	0.7645 and 0.7431
Unit cell dimensions	$a = 2.1170(3)$ nm $\alpha = 90^\circ$ $b = 1.1278(1)$ nm $\beta = 90^\circ$ $c = 1.8956(2)$ nm $\gamma = 90^\circ$	Refinement method	Full-matrix least-squares on F^2
Volume, Z	4.5258(9) nm ³ , 4	Data/restraints/parameters	5618/8/599
Density (calculated)	1.382 g/cm ³	Goodness-of-fit on F^2	0.927
Absorption coefficient	0.532 mm ⁻¹	Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0354$, $wR_2 = 0.0751$
$F(000)$	1972	R indices (all data)	$R_1 = 0.0510$, $wR_2 = 0.0795$
Crystal size (mm)	0.56 × 0.54 × 0.52	Absolute structure parameter	0.013(13)
		Extinction coefficient	0.00130(16)
		Largest diff. Peak and hole	340 and -228 e/nm ³

Table 2 Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{nm}^2 \times 10$) for $\text{Co}(\text{PMBP-tsc})_2 \cdot 2\text{DMF} \cdot 2\text{H}_2\text{O}$

Atoms	X	Y	Z	U(eq)	Atoms	X	Y	Z	U(eq)
Co	3390(1)	5156(1)	6641(1)	28(1)	C(18)	2227(2)	6645(4)	4783(2)	43(1)
S(1)	3752(1)	3722(1)	7314(1)	39(1)	C(19)	2356(2)	5551(3)	7633(2)	32(1)
S(2)	3095(1)	6219(1)	7562(1)	38(1)	C(20)	2329(2)	3751(3)	6179(2)	30(1)
O(1)	3040(1)	6319(2)	6000(1)	38(1)	C(21)	1662(2)	3318(3)	6228(2)	34(1)
O(2)	3707(1)	4334(2)	5810(1)	39(1)	C(22)	1529(2)	2273(3)	6573(3)	50(1)
N(1)	4865(2)	3868(4)	7943(2)	51(1)	C(23)	919(2)	1814(4)	6552(3)	66(1)
N(2)	4621(1)	5396(3)	7200(2)	36(1)	C(24)	451(2)	2384(5)	6195(3)	70(1)
N(3)	4222(1)	5852(2)	6680(2)	31(1)	C(25)	579(2)	3417(5)	5867(3)	72(2)
N(4)	3243(1)	7541(3)	5049(2)	35(1)	C(26)	1186(2)	3902(4)	5878(2)	53(1)
N(5)	3743(1)	8169(3)	4752(2)	44(1)	C(27)	3335(2)	3650(3)	5470(2)	34(1)
N(6)	1974(2)	5940(3)	8162(2)	45(1)	C(28)	2695(2)	3343(3)	5589(2)	35(1)
N(7)	2148(1)	4730(2)	7214(1)	32(1)	C(29)	2549(2)	2526(4)	5043(2)	45(1)
N(8)	2559(1)	4461(2)	6659(2)	29(1)	C(30)	1942(2)	1919(5)	4856(3)	69(2)
N(9)	3527(1)	3017(3)	4894(2)	42(1)	C(31)	4132(2)	2971(4)	4575(2)	44(1)
N(10)	3040(2)	2325(3)	4635(2)	54(1)	C(32)	4307(2)	1939(4)	4225(2)	58(1)
C(1)	4465(2)	4351(3)	7496(2)	37(1)	C(33)	4890(2)	1907(6)	3881(3)	74(2)
C(2)	4463(2)	6636(3)	6247(2)	32(1)	C(34)	5294(2)	2863(6)	3913(3)	79(2)
C(3)	5146(2)	6973(3)	6308(2)	36(1)	C(35)	5124(2)	3853(5)	4270(3)	70(1)
C(4)	5327(2)	7950(3)	6692(3)	49(1)	C(36)	4533(2)	3933(4)	4601(2)	55(1)
C(5)	5952(2)	8291(4)	6720(3)	58(1)	O(4)	5829(1)	5546(3)	7769(2)	63(1)
C(6)	6392(2)	7659(5)	6358(2)	63(1)	N(12)	6793(2)	5238(3)	8237(2)	57(1)
C(7)	6228(2)	6669(5)	5980(2)	63(1)	C(40)	6636(3)	4052(5)	8477(4)	110(3)
C(8)	5599(2)	6320(4)	5955(2)	45(1)	C(41)	7412(2)	5669(5)	8404(3)	82(2)
C(9)	3428(2)	6918(3)	5627(2)	32(1)	C(42)	6368(2)	5868(4)	7904(2)	51(1)
C(10)	4080(1)	7147(3)	5711(2)	32(1)	O(5)	4772(2)	1389(3)	8160(2)	80(1)
C(11)	4235(2)	7938(3)	5146(2)	40(1)	O(6)	6085(2)	1107(3)	8258(2)	73(1)
C(12)	4859(2)	8487(4)	4956(3)	65(1)	O(3)	4148(2)	470(5)	7465(3)	132(2)
C(13)	2631(2)	7606(3)	4745(2)	35(1)	N(11)	3466(2)	63(4)	6577(4)	81(1)
C(14)	2457(2)	8626(4)	4392(2)	47(1)	C(37)	3822(5)	812(6)	6274(5)	185(5)
C(15)	1870(2)	8684(4)	4061(2)	59(1)	C(38)	2866(3)	372(8)	6294(5)	148(4)
C(16)	1470(2)	7725(5)	4098(2)	66(1)	C(39)	3678(3)	676(6)	7113(4)	96(2)
C(17)	1635(2)	6706(4)	4453(2)	52(1)					

Table 3 Selected bond lengths (nm) selected bond angles ($^\circ$) for $\text{Co}(\text{PMBP-tsc})_2 \cdot 2\text{DMF} \cdot 2\text{H}_2\text{O}$

Bond lengths (nm)					
Co—N(8)	0.1926(2)	Co—N(3)	0.1931(2)	Co—O(1)	0.1937(2)
Co—S(1)	0.2197(1)	Co—S(2)	0.2208(1)	S(1)—C(1)	0.1703(3)
Co—O(2)	0.1948(2)	S(2)—C(19)	0.1741(3)	N(1)—C(1)	0.1316(5)
O(1)—C(9)	0.1278(4)	O(2)—C(27)	0.1277(4)	N(3)—C(2)	0.1310(4)
N(2)—C(1)	0.1346(4)	N(2)—N(3)	0.1396(4)	N(7)—C(19)	0.1298(4)
N(4)—C(9)	0.1358(4)	N(4)—N(5)	0.1394(4)	N(9)—C(27)	0.1367(4)
N(5)—C(11)	0.1307(4)	N(6)—C(19)	0.1361(4)	N(10)—C(29)	0.1314(4)
N(7)—N(8)	0.1498(4)	N(8)—C(20)	0.1308(4)	C(9)—C(10)	0.1412(4)
N(9)—N(10)	0.1384(4)	C(10)—C(11)	0.1431(5)	C(20)—C(28)	0.1436(4)
C(2)—C(10)	0.1423(4)	C(20)—C(21)	0.1496(4)	C(28)—C(29)	0.1420(5)
C(27)—C(28)	0.1416(5)				

Continued

Bond angles (°)					
N(8)-Co-N(3)	176.81(1)	N(8)-Co-O(1)	86.37(1)	N(3)-Co-O(1)	95.61(1)
N(8)-Co-O(2)	97.81(1)	N(3)-Co-O(2)	84.81(1)	O(1)-Co-O(2)	86.89(1)
N(8)-Co-S(1)	90.52(8)	N(3)-Co-S(1)	87.66(9)	O(1)-Co-S(1)	175.24(8)
O(2)-Co-S(1)	89.97(8)	N(8)-Co-S(2)	87.02(9)	N(3)-Co-S(2)	90.44(9)
O(1)-Co-S(2)	91.14(8)	O(2)-Co-S(2)	174.65(8)	S(1)-Co-S(2)	92.30(4)
C(1)-S(1)-Co	96.89(1)	C(19)-S(2)-Co	94.66(1)	C(9)-O(1)-Co	117.3(2)
C(27)-O(2)-Co	118.8(2)	C(1)-N(2)-N(3)	117.9(3)	C(2)-N(3)-N(2)	117.1(2)
C(2)-N(3)-Co	127.4(2)	N(2)-N(3)-Co	115.4(2)	C(9)-N(4)-N(5)	111.6(3)
C(19)-N(7)-N(8)	113.8(2)	N(5)-N(4)-C(13)	120.2(3)	C(11)-N(5)-N(4)	105.8(3)
N(7)-N(8)-Co	119.6(2)	C(20)-N(8)-N(7)	115.1(2)	C(20)-N(8)-Co	125.3(2)
N(1)-C(1)-N(2)	118.2(3)	C(27)-N(9)-N(10)	110.9(3)	C(29)-N(10)-N(9)	106.4(3)
N(3)-C(2)-C(10)	119.9(3)	N(1)-C(1)-S(1)	121.8(3)	N(2)-C(1)-S(1)	119.9(2)
C(9)-C(10)-C(2)	124.3(3)	C(9)-C(10)-C(11)	104.7(3)	N(4)-C(9)-C(10)	106.2(3)
N(5)-C(11)-C(10)	111.7(3)	N(7)-C(19)-S(2)	124.5(2)	C(2)-C(10)-C(11)	131.0(3)
N(7)-C(19)-N(6)	118.6(3)	O(2)-C(27)-C(28)	131.1(3)	N(6)-C(19)-S(2)	116.9(3)
N(8)-C(20)-C(28)	122.5(3)	C(27)-C(28)-C(20)	124.2(3)	N(9)-C(27)-C(28)	106.5(3)
C(27)-C(28)-C(29)	104.5(3)	C(29)-C(28)-C(20)	131.1(3)	N(10)-C(29)-C(28)	111.7(3)

Results and discussion

IR spectra

A broad absorption band at 2537 cm^{-1} in the free ligand spectrum is attributed to $\nu_{\text{O-H}}$,¹⁵ which suggests the existence of the white PMBP-tsc in the enol form in the solid state and the presence of intermolecular hydrogen bonding.¹⁶ However, the band has disappeared in the spectrum of the complex and a new absorption band assigned to $\text{C}=\text{O}$ has appeared at 1593 cm^{-1} , indicating the involvement of the oxygen atom of carbonyl group in the pyrazolone-ring in bonding to the Co(II) .

The strong IR band appearing at 1577 cm^{-1} for $\text{C(2)}=\text{N(3)}$ in the free ligand has been observed to shift towards lower wave number by 14 cm^{-1} in the spectrum of the complex due to the involvement of the azomethine nitrogen in bonding. The bands at $3341\text{ [N(2)-H] cm}^{-1}$ and 836 (C=S) cm^{-1} in the free ligand have disappeared in the complex, with appearance of new bands at 1593 cm^{-1} and 760 cm^{-1} assigned to $(\text{S}-\text{C}=\text{N})$ ¹⁷ and $(\text{C}-\text{S})$,¹⁸ respectively. These results indicate that thione form of the ligand isomerizes to the thiol form in the reaction. Then the reagent loses a proton from its tautomeric thiol form and acts as a singly charged tridentate ligand coordinating to cobalt(II) via the mercapto S.

Structure of $\text{Co(PMBP-tsc)}_2 \cdot 2\text{DMF} \cdot 2\text{H}_2\text{O}$

The molecular structure and packing diagram of the complex are shown in Fig. 1 and Fig. 2, respectively. As expected, the cobalt(II) atom is octahedrally coordinated by the two tridentate ONS-ligands. Two ligands chelate to Co^{2+} ion with the azomethine nitrogen atoms positioning *trans* to each other. But the oxygen and sulphur atoms do not lie mutually in the *trans* positions. The $\text{Co}-\text{S(2)}$, $\text{Co}-\text{O(2)}$ are 0.2208 nm , 0.1948 nm . The S(2)-Co-O(2) angle is 174.65° . The

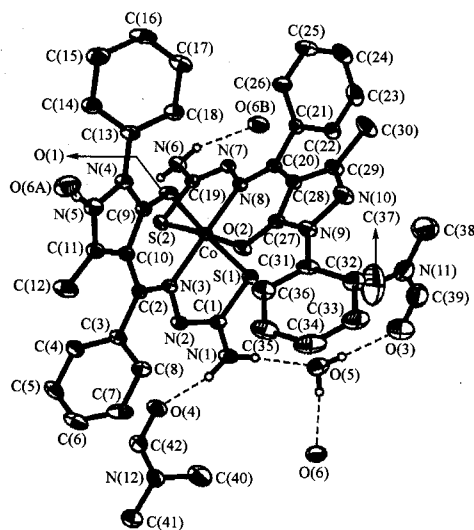


Fig. 1 Molecular structure of the title compound.

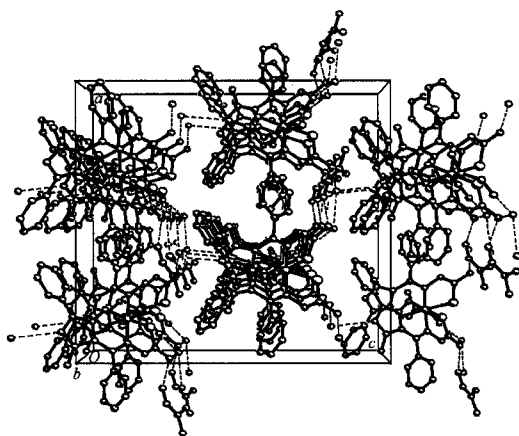


Fig. 2 Unit cell packing diagram of the title compound.

$S(1)-Co-S(2)$ (92.30°), $S(2)-Co-N(8)$ (87.02°), $N(8)-Co-O(2)$ (97.81°) angular distributions indicate that the coordination polyhedron is distorted.

Useful information can be derived by comparing the structure of the ligand in the free state and in the Co(II) complex, some bond lengths and bond angles were changed. As far as bond distances around atoms C(1), C(19) in the Co(II) complex are concerned, the changes may be the most obvious. The bond distances of C(1)—S(1) and C(19)—S(2) are 0.1703 nm and 0.1741 nm in the complex respectively, slightly longer than that of C—S (0.1672 nm)¹⁵ in the ligand. While

the bond lengths of C(1)—N(1) and C(19)—N(7) in the title compound are 0.1316 nm and 0.1298 nm respectively, slightly shorter than the corresponding C—N (0.1356 nm)¹⁵ distance in the ligand. These changes indicate the involvement of sulfur atom in bonding. The result is further supported by IR spectroscopic studies of the free ligand and the complex molecule.

Coordination to the metal center causes inter-plane twisting, the dihedral angles between them are listed in Table 4. The plane III in one ligand is nearly perpendicular to the planes II and IV, while the planes II and IV are nearly coplaner. So do the planes I and V and those of the other ligands contained in the structure.

It is interesting that two DMF molecules and two water molecules are contained in the structure of the complex. The DMF molecules are linked to the complex by the hydrogen bonds [$N(1)\cdots O(4)$, 0.2806(5) nm; $N(1)\cdots O(5)$, 0.2835(6) nm; $O(5)\cdots O(3)$, 0.2795(7) nm]. Two water molecules were bridged through hydrogen bond [$O(5)\cdots O(6)$, 0.2795(5) nm]. The hydrogen bonds [$N(6)\cdots O(6)$, 0.2984(5) nm; $N(5)\cdots O(6)$, $N(1)\cdots O(5)$, $O(5)\cdots O(6)$] exist between the neighboring molecules. Thus three-dimensional hydrogen-bond networks assembled the moieties into a supramolecule as shown in Fig. 2. All above hydrogen bonds in this structure may bring additional stabilization for the complex.

Table 4 Dihedral angles ($^\circ$) for selected planes of $Co(PMBP-tsc)_2 \cdot 2DMF \cdot 2H_2O$

Plane	Atom defining plane	Dihedral angle			
		I	II	III	IV
I	C(13), C(14), C(15), C(16), C(17), C(18)				
II	N(4), N(5), C(9), C(10), C(11)	30.4			
III	C(3), C(4), C(5), C(6), C(7), C(8)	65.6	90.6		
IV	Co, O(1), C(9), C(10), C(2), N(3)	23.8	7.2	83.6	
V	Co, S(1), C(1), N(2), N(3)	9.7	20.8	74.1	14.5
Plane	Atom defining plane	Dihedral angle			
		I'	II'	III'	IV'
I'	C(31), C(32), C(33), C(34), C(35), C(36)				
II'	N(9), N(1), C(27), C(28), C(29)	27.5			
III'	C(21), C(22), C(23), C(24), C(25), C(26)	65.2	92.8		
IV'	Co, O(2), C(27), C(28), C(20), N(8)	29.3	3.1	90.0	
V'	Co, S(2), C(19), N(7), N(8)	24.8	5.6	92.7	50.3

Supplementary data

Supplementary data are available from the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK. The deposition number is 155181.

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