

5,5'-二硫双(2-硝基苯甲酸)构筑的钴配合物的合成与晶体结构

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摘要: 以柔性配体 5,5'-二硫双(2-硝基苯甲酸)为主配体, 与含氮辅助配体在水热条件下合成了 2 个 Co(II)配合物 $[\text{Co}(\text{dtb})(\text{bpp}) \cdot \text{H}_2\text{O}]_n$ (**1**), $[\text{Co}(\text{dtb})(\text{phen}) \cdot 2\text{H}_2\text{O}]_n$ (**2**) (H_2dtb =5,5'-二硫双(2-硝基苯甲酸), phen =1,10-菲咯啉, bpp =1,3-二(4-吡啶)-丙烷), 通过 X-射线单晶衍射、X-射线粉末衍射、红外光谱、热重分析和元素分析方法对其进行表征。结构测试表明 **1** 为正交晶系, $Pbca$ 空间群, 晶胞参数为 $a=1.014\ 9(2)\ \text{nm}$, $b=2.215\ 9(5)\ \text{nm}$, $c=2.529\ 1(6)\ \text{nm}$, $V=5.688(2)\ \text{nm}^3$, $D_c=1.564\ \text{g} \cdot \text{cm}^{-3}$, $Z=8$ 。**2** 为三斜晶系, $P\bar{1}$ 空间群, 晶胞参数为 $a=0.740\ 1(3)\ \text{nm}$, $b=1.074\ 2(4)\ \text{nm}$, $c=1.783\ 7(6)\ \text{nm}$, $\alpha=98.452(7)^\circ$, $\beta=99.816(7)^\circ$, $\gamma=98.979(7)^\circ$, $V=1.358\ 0(8)\ \text{nm}^3$, $D_c=1.637\ \text{g} \cdot \text{cm}^{-3}$, $Z=2$ 。化合物 **1** 为三维网络结构, 并包含单股双轴假螺旋结构。配合物 **2** 为一维链状结构, 相邻一维链之间通过氢键作用形成二维超分子网络结构。

关键词: 双硫键配体; 配位聚合物; 5,5'-二硫双(2-硝基苯甲酸)

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Syntheses and Crystal Structures of Co(II) Coordination Polymers Constructed from a Flexible 5,5'-Dithiobis(2-nitrobenzoic acid) Ligand

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Abstract: Two complexes, $[\text{Co}(\text{dtb})(\text{bpp}) \cdot \text{H}_2\text{O}]_n$ (**1**), $[\text{Co}(\text{dtb})(\text{phen}) \cdot 2\text{H}_2\text{O}]_n$ (**2**), (H_2dtb =5,5'-dithiobis(2-nitrobenzoic acid), bpp =1,3-bis(4-pyridyl)propane), have been constructed by H_2dtb ligand and Co(II) salt in the presence of assistant N-containing ligands under hydrothermal conditions. The structure parameters were analyzed as follows: **1**, orthorhombic, $Pbca$, $a=1.014\ 9(2)\ \text{nm}$, $b=2.215\ 9(5)\ \text{nm}$, $c=2.529\ 1(6)\ \text{nm}$, $V=5.688(2)\ \text{nm}^3$, $D_c=1.564\ \text{g} \cdot \text{cm}^{-3}$, $Z=8$. **2**, triclinic, $P\bar{1}$, $a=0.740\ 1(3)\ \text{nm}$, $b=1.074\ 2(4)\ \text{nm}$, $c=1.783\ 7(6)\ \text{nm}$, $\alpha=98.452(7)^\circ$, $\beta=99.816(7)^\circ$, $\gamma=98.979(7)^\circ$, $V=1.358\ 0(8)\ \text{nm}^3$, $D_c=1.637\ \text{g} \cdot \text{cm}^{-3}$, $Z=2$. Complex **1** displays a 3D non-interpenetrating structure containing a fascinating meso-helices structure with two spiral shafts in one helical chain. X-Ray structure analysis of **2** shows a 1D chain formed a 2D supramolecular network by hydrogen bonding. Solid-state properties of thermal stability and X-ray powder diffractions for these crystalline materials have been investigated. CCDC: 929076, **1**; 929077, **2**.

Key words: disulfide ligand; coordination polymer; 5,5'-dithiobis(2-nitrobenzoic acid)

The currently impetus for the design and fabrication of novel metal-organic frameworks (MOFs) is rooted not only in their fascinating topological structures but also from their promising properties and

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great potential applications in the fields of gas storage, magnetism, and optical properties, and so on^[1-4]. As is known to all, the prediction of coordination network is still subjective and cannot be generalized by some factors, such as solvent, temperature, and metal/ligand nature. It has been observed that organic ligands play crucial roles for the designed synthesis of some interesting coordination networks, such as the donating type, the flexibility, and the geometry of the organic ligands^[5]. In contrast to rigid organic ligands, the coordination networks constructed from flexible ligands are relatively underdeveloped, that's because it difficult to predict either the composition or the network topology constructed from a flexible ligand due to the conformational freedom in the assembly process^[6]. However, the special conformation and coordination functionality of the flexible ligand might generate some unprecedented coordination frameworks. In this regard, flexible disulfide derivatives bearing -S-S- spacers have attracted considerable interest, because they can afford more rich structure information compared with rigid ligands^[7-9]. Nevertheless, it is still a challenge to predict and control the ligand reactions toward our target products, and more knowledge of this field is needed^[8]. In this report, we focused our attention on utilizing a flexible disulfide derivative of the carboxylate ligand 5,5'-dithiobis (2-nitrobenzoic acid) (H_2dtb) adopting a twist conformation with the C-S-S-C torsion angles in aromatic carboxylates are known to be generally about 20° to 90° and the axial chirality can potentially generate the *M*- and *P*-enantiomers in chiral crystal engineering, which may facilitate the formation of a helical coordination polymer^[10-12].

On the other hand, the introduction of additional N-donor ligands to the synthetic systems is of great potential to construct higher-dimensional supramolecular networks and novel topology^[13-16]. As for the N-donor ligands, which are introduced into M-dtb system may bring on new structures and allow fine-tuning of the structural motif of these metal-organic compounds. Employing this strategy, we have successfully gained two new coordination polymers namely $\{[Co(dtb)(bpp) \cdot$

$H_2O]\}_n$ (**1**) and $\{[Co(dtb)(phen) \cdot 2H_2O]\}_n$ (**2**) by utilizing the H_2dtb ligand in the presence of two assistant ligands (bpp, phen) and Co (II) salt. The crystal structures of two compounds, along with the investigation on the effect of coordination modes of H_2dtb and neutral assistant ligands on the ultimate framework, will be presented and discussed. In addition, TG analysis, and XRPD of compounds **1** and **2** have been discussed.

1 Experimental

1.1 Materials and physical measurements

The reagents were used directly as supplied commercially without further purification. Elemental analyses (C, H, N) were determined with a Perkin-Elmer model 240C automatic instrument. Infrared spectra on KBr pellets were recorded on a Bruker Equinox-55 spectrometer in the range of 4 000~400 cm^{-1} . Luminescence spectra for the solid samples were investigated with a Hitachi F-4500 fluorescence spectrophotometer. The X-ray powder diffraction pattern was recorded with a Rigaku D/Max 3III diffractometer. Thermal analysis was determined with a Netzsch STA 449C microanalyzer under flowing N_2 atmosphere at a heating rate of 10 $^{\circ}C \cdot min^{-1}$.

1.2 Synthesis of $\{[Co(dtb)(bpp) \cdot H_2O]\}_n$ (**1**)

Compound **1** was obtained by the reaction of $Co(OAc)_2 \cdot 4H_2O$ (0.025 g, 0.1 mmol), H_2dtb (0.039 g, 0.1 mmol), bpp (0.018 g, 0.1 mmol), ethanol (2.0 mL) and distilled water (15.0 mL) under hydrothermal conditions (at 150 $^{\circ}C$ for 6 days and cooled to room temperature with a rate of 5 $^{\circ}C \cdot h^{-1}$). The pH was adjusted to 7 with NaOH (1 mol $\cdot L^{-1}$), and the pH adjustment was done before the reaction started. Red crystal of **1** was collected in 55% yield. Anal. Calcd. (%) for $C_{27}H_{22}CoN_4O_9S_2$: C, 48.03 H, 3.63; N, 9.08. Found(%): C, 51.09; H, 3.51; N, 9.36. IR (KBr, cm^{-1}): 3 448(w), 3 208(w), 3 042(w), 1 648(s), 1 561(s), 1 520(s), 1 332(s), 1 135(w), 1 092(w), 1 045(w), 930(w), 855 (w), 822(m), 760(w), 736(m), 658(w).

1.3 Synthesis of $\{[Co(dtb)(phen) \cdot 2H_2O]\}_n$ (**2**)

Compound **2** was synthesized in an analogous procedure to **1** except that bpp was replaced by phen,

giving red block X-ray-quality crystals in a 60% yield. Anal. Calcd.(%) for $C_{26}H_{18}CoN_4O_{10}S_2$: C, 48.15; H, 3.07; N, 8.08. Found (%): C, 50.09; H, 2.74; N, 9.71. IR (KBr, cm^{-1}): 3 421 (w), 3 234(w), 3 042(w), 1 626 (s), 1 583(s), 1 522(s), 1 421(s), 1 368(s), 1 136(w), 1 096 (w), 1 058(w), 882(w), 845(m), 790(w), 760(m), 688(m).

1.4 Crystallographic data collection and refinement

Single-crystal X-ray diffraction analyses of the compounds **1** and **2** were carried out on a Bruker SMART APEX II CCD diffractometer equipped with a graphite monochromated Mo $K\alpha$ radiation ($\lambda=0.071\ 073$ nm) by using φ/ω scan technique at room temperature.

The structures were solved using direct methods and successive Fourier difference synthesis (SHELXS-97)^[17], and refined using the full-matrix least-squares method on F^2 with anisotropic thermal parameters for all non-hydrogen atoms (SHELXL-97)^[18]. All non-hydrogen atoms were refined anisotropically. All solvent hydrogen atoms were located in successive different Fourier Maps and the other hydrogen atoms were treated as riding method. The crystallographic data for **1** and **2** are listed in Table 1, and selected bond lengths and angles are listed in Table 2. The hydrogen bond geometry of complex **2** is listed in Table 3.

CCDC: 929076, **1**; 929077, **2**.

Table 1 X-Ray crystal structure data for compounds **1** and **2**

Complex	1	2
Empirical formula	$C_{27}H_{22}CoN_4O_9S_2$	$C_{26}H_{18}CoN_4O_{10}S_2$
Formula weight	669.54	669.49
Crystal system	Orthorhombic	Triclinic
Space group	$Pbca$	$P\bar{1}$
a / nm	1.014 9(2)	0.740 1(3)
b / nm	2.215 9(5)	1.074 2(4)
c / nm	2.529 1(6)	1.783 7(6)
α / ($^\circ$)	90	98.452(7)
β / ($^\circ$)	90	99.816(7)
γ / ($^\circ$)	90	98.979(7)
V / nm^3	5.688(2)	1.358 0(8)
Z	8	2
D_c / ($g \cdot cm^{-3}$)	1.564	1.637
μ / mm^{-1}	0.811	0.852
$F(000)$	2 744.0	682.0
θ range / ($^\circ$)	1.84~25.10	1.18~25.10
Reflections collected	27 318	6 841
Reflections / unique	5 047 / 1 782	4 699 / 2 107
R_{int}	0.164 6	0.045 2
Goodness-of-fit on F^2	1.021	1.022
Final R^a indices [$I > 2\sigma(I)$]	$R_1=0.063\ 8$, $wR_2=0.182\ 6$	$R_1=0.057\ 8$, $wR_2=0.172\ 9$

$$^a R_1 = \sum (|F_o| - |F_c|) / \sum |F_o|; wR_2 = [\sum w(F_o^2 - F_c^2)^2 / \sum w(F_o^2)^2]^{1/2}.$$

Table 2 Selected bond lengths (nm) and angles ($^\circ$) for compound **1** and **2**

1					
Co(1)-O(9)	0.208 6(4)	Co(1)-O(1)	0.211 8(5)	Co(1)-O(5)a	0.212 8(5)
Co(1)-N(2)	0.213 9(6)	Co(1)-O(6)b	0.214 2(5)	Co(1)-N(1)	0.217 4(6)
O(9)-Co(1)-O(1)	83.07(18)	O(9)-Co(1)-O(5)a	162.78(19)	O(1)-Co(1)-O(5)a	82.97(19)
O(9)-Co(1)-N(2)	87.1(2)	O(1)-Co(1)-N(2)	90.3(2)	O(5)a-Co(1)-N(2)	83.0(2)

Continued Table 2

O(9)-Co(1)-O(6)b	96.90(18)	O(1)-Co(1)-O(6) ^b	175.20(18)	O(5)a-Co(1)-O(6)b	96.17(18)
N(2)-Co(1)-O(6)b	84.9(2)	O(9)-Co(1)-N(1)	99.1(2)	O(1)-Co(1)-N(1)	95.3(2)
O(5)a-Co(1)-N(1)	92.2(2)	N(2)-Co(1)-N(1)	172.2(3)	O(6)b-Co(1)-N(1)	89.5(2)
2					
Co(1)-N(2)	0.215 5(6)	Co(1)-O(7)b	0.205 5(5)	Co(1)-O(1)	0.205 6(5)
Co(1)-N(1)	0.211 1(6)	Co(1)-O(9)	0.213 9(5)	Co(1)-O(10)	0.214 9(4)
O(7)b-Co(1)-O(1)	94.12(19)	O(7)b-Co(1)-N(1)	93.7(2)	O(1)-Co(1)-N(1)	167.9(2)
O(7)b-Co(1)-O(9)	92.4(2)	O(1)-Co(1)-O(9)	79.96(19)	N(1)-Co(1)-O(9)	90.4(2)
O(7)b-Co(1)-O(10)	89.11(18)	O(1)-Co(1)-O(10)	91.11(18)	N(1)-Co(1)-O(10)	98.3(2)
O(9)-Co(1)-O(10)	171.03(19)	O(7)b-Co(1)-N(2)	170.5(2)	O(1)-Co(1)-N(2)	95.1(2)
N(1)-Co(1)-N(2)	77.6(2)	O(9)-Co(1)-N(2)	91.3(2)	O(10)-Co(1)-N(2)	88.63(19)

Symmetry code: For **1**: a: $-x+1, y+1/2, -z+1/2$; b: $-x+1/2, y+1/2, z$; For **2**: b: $x+1, y+1, z$.

Table 3 Hydrogen-bonding geometries for compound **2**

D-H...A	<i>d</i> (D-H) / nm	<i>d</i> (H...A) / nm	<i>d</i> (D...A) / nm	∠DHA / (°)
O(9)-H(9WB)...O(2)a	0.074	0.204	0.273 7(7)	157
O(10)-H(10A)...O(8)a	0.085	0.191	0.267 7(7)	149
O(10)-H(10B)...O(2)	0.085	0.206	0.278 9(7)	143

Symmetry code: a: $1+x, y, z$.

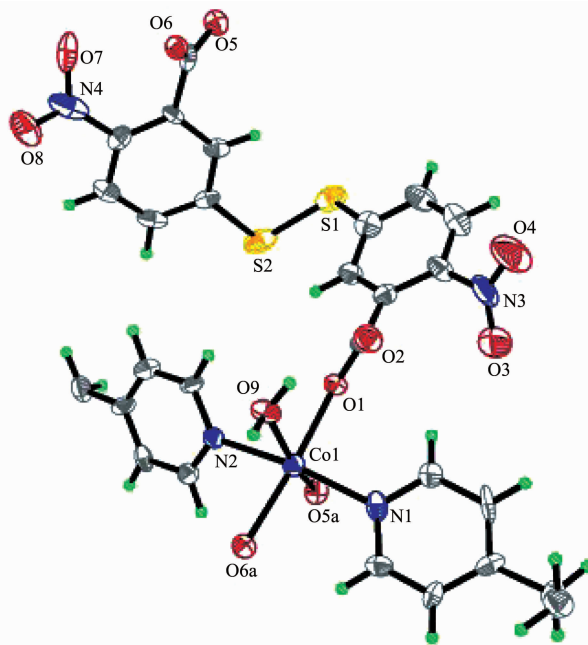
2 Results and discussion

2.1 Description of the structure of $[\text{Co}(\text{dtb})(\text{bpp}) \cdot \text{H}_2\text{O}]_n$ (**1**)

In **1**, the metal Co(II) center possesses a distorted octahedral geometry (Fig.1), being linked by four oxygen atoms from three carboxylic groups of dtb^{2-} ligand and one coordinated water in the equatorial sites, and two nitrogen atoms from two bpp ligands in the axial positions. The Co-O and Co-N bond distances are in the range 0.208 6(4)~0.214 2(5) nm and 0.213 9(6)~0.217 4(6) nm, which are in agreement with those reported for Co(II) complexes^[19]. The dihedral angle for the two phenyl planes of the dtb ligand is 78.82°. Completely deprotonated dtb^{2-} ligands perform as monodentate/bi(monodentate) bridging mode to join up Co(II) atoms to propagate into a 2D corrugated network (Fig.2).

Furthermore, in the 2D corrugated network, there exists a fascinating meso-helices based on the dtb^{2-} ligand and Co(II) ions with two spiral shafts in one helical chain (Fig.3). In previous work, our group successfully constructed a 3D metal-organic framework by dtb^{2-} ligand, in which exists the first example of

metal-organic frameworks with four spiral shafts in one helical chain and can be named a tetraflexural helix^[20]. As a V-shaped aromatic polycarboxylate, H_2dtb features the bent orientation between two



Symmetry code: a: $1-x, 0.5+y, 0.5-z$; Thermal ellipsoids were drawn at the 30% level

Fig.1 Local coordination environment of the Co(II) ion in compound **1**

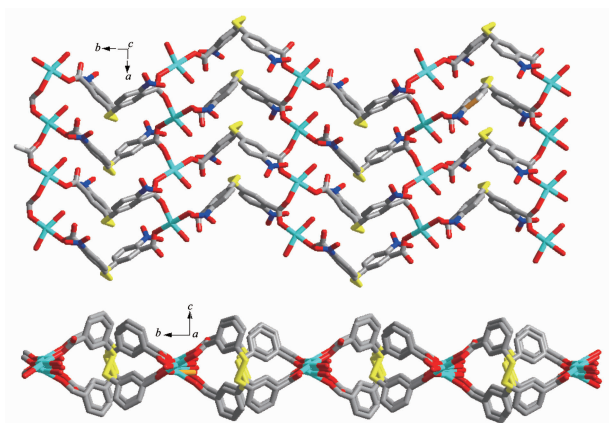


Fig.2 2D corrugated network in **1** from the different direction

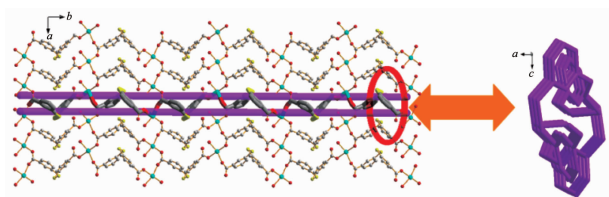


Fig.3 Meso-helices based on the dtb ligand and Co(II) ions with two spiral shafts in **1**

terminal coordinated groups and is considered as a nice candidate for the construction of novel helical coordination polymers. In addition, for the view of coordinated behavior of bpp in **1**, the long bpp ligands bond the Co(II) atoms to form a 2D sheet (Fig.4). Ultimately, the two kinds of 2D networks in **1** interweave with each other to form a 3D non-interpenetrating structure (Fig.5).

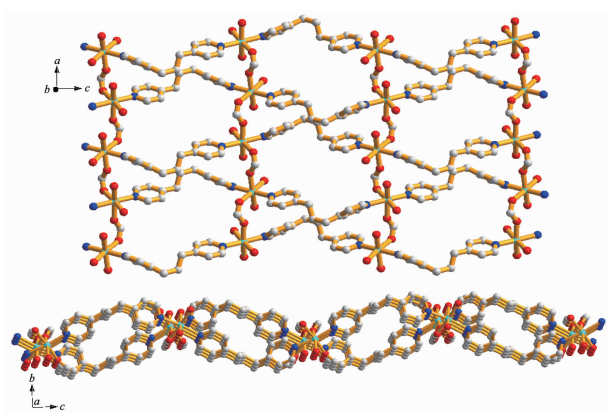


Fig.4 2D network constructed by bpp ligand and Co(II) ions in **1**

2.2 Description of the structure of $[\text{Co}(\text{dtb})(\text{phen}) \cdot 2\text{H}_2\text{O}]_n$ (**2**)

The asymmetric unit contains one Co(II) ion, one

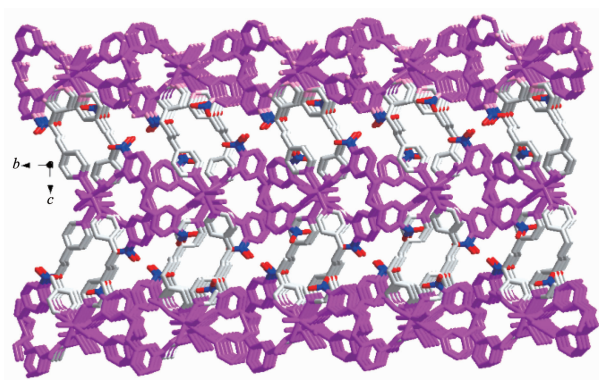
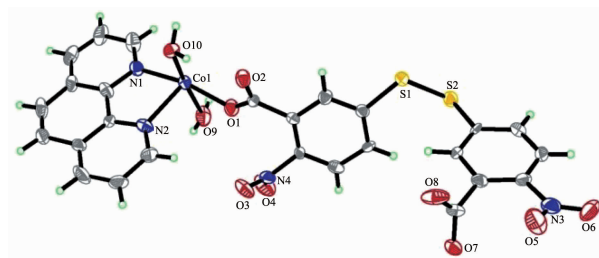


Fig.5 Perspective view of the 3D network in **1**

dtb^{2-} ligand, one phen ligand, and two coordinated water molecules (Fig.6). Each Co(II) ion is primarily coordinated by two oxygen atoms from two dtb^{2-} ligands and two nitrogen atoms from one chelating phen ligand in the equator plane and two oxygen atoms from coordinated water molecules occupying the axial position to furnish a distorted octahedral geometry (Co-O 0.205 5(5)~0.214 9(4) nm, Co-N 0.211 1(6)~0.215 5(6) nm). The dihedral angle for the two phenyl planes of the dtb^{2-} ligand is 76.03° . The carboxylate group of dtb^{2-} ligand coordinates to Co(II) ion with a bridging bis(monodentate) mode to produce a 1D chain (Fig.7), which is formed a 2D supramolecular network by hydrogen bonding between the $-\text{COO}^-$ group of dtb^{2-} ligands and the coordinated waters (Fig.8).



Thermal ellipsoids were drawn at the 30% level

Fig.6 Local coordination environment of the Co(II) ion in compound **2**

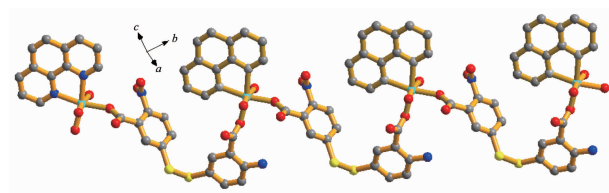
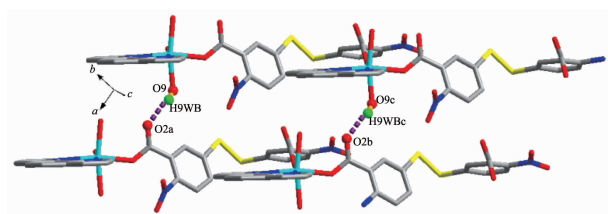


Fig.7 Perspective views of the 1D chain structure of **2**



Symmetry code: a: $1+x, y, z$; b: $x, -1+y, z$; c: $-1+x, -1+y, z$

Fig.8 2D supramolecular network formed through hydrogen bonding in **2**

2.2 Comparison of structures of coordination polymers

In **1**, the dtb^{2-} ligand serves as a $(\kappa^1-\kappa^1)-\kappa^1-\mu_3$ coordination mode and connects Co(II) ions to form a 2D corrugated network. In **2**, the carboxylate group of the dtb^{2-} ligand adopts a $(\kappa^1-\kappa^1)-\mu_2$ bridging mode, being linked by Co(II) atoms to bring on a 1D chain. The results described above show that the dtb^{2-} ligand can afford relatively various coordination modes to meet the coordination requirements of the center metal ions. However, for the complexes without N-donor auxiliary ligands, they may favor the formation of lower dimensional structures. In comparison, when the N-donor auxiliary ligands are introduced in the system, the drastic changes are observed from low-dimension to high-dimension in the structures [21]. Furthermore, compared to rigid carboxylate ligands, the flexible S-S bond in the dtb^{2-} ligand can rotate freely and the dihedral angle of the two phenyl rings is changeable in a considerable range. Hence, the V-shaped configuration of dtb^{2-} ligand is considered as a

good candidate for the construction of helix, because of its twist conformation with a C-S-S-C torsion angle of *ca.* 90° and axial chirality that potentially generate the *M*- and *P*- enantiomers in chiral crystal engineering, similar to the relative literature [22]. Furthermore, in compound **1**, the bpp ligand acting as the pillars extend the 2D Co(II)-dtb network to result in the formation of the 3D framework. This means that the dimensionality of the compound can increase through the linkage of bpp. On the other hand, the chelating N-donor ligand (phen) was employed as the starting reactant, which can not extend above the 1D chain. This implied that the steric hindrance and chelating effects of these terminal ligands can constrain the prolongation of the framework and result in the formation of lower dimensionality [23]. Although the above-mentioned factors work together to affect the structures of compounds, it is difficult to control and separate them, because all the variable factors cannot be accurately forecasted at this stage. Therefore, a comprehensive research is required to be developed and a rational design would be a long-range challenge.

2.3 XRPD and thermal stability analyses

The XRPD patterns of complexes **1** and **2** reveal that all compounds are pure single phase and are also of completely identical molecular structures, corresponding to the simulated results from the single-crystal X-ray data (Fig.9).

To study the thermal stabilities of these

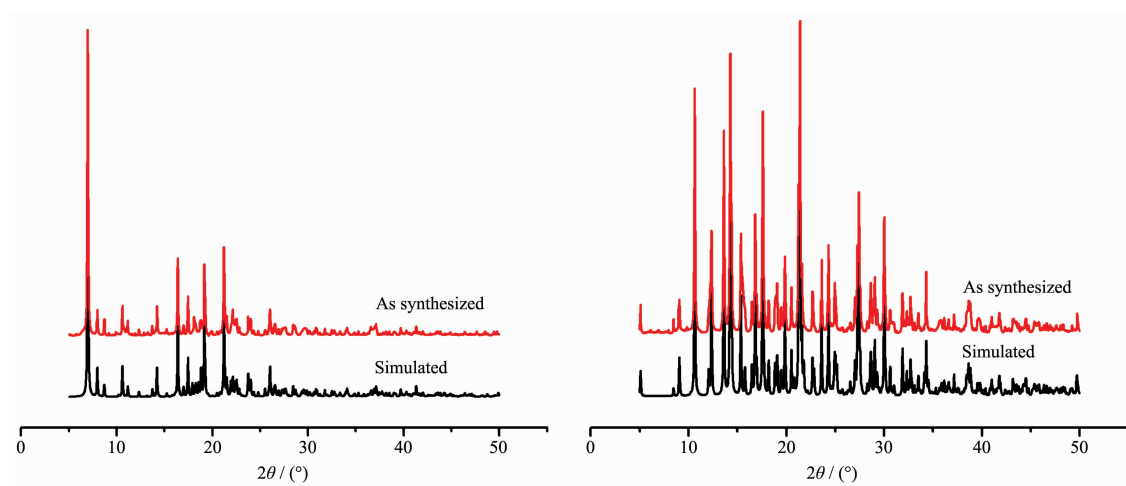


Fig.9 XRPD patterns of complexes **1** (left) and **2** (right)

complexes, thermal gravimetric analysis (TGA) of complexes **1** and **2** was carried out under N₂ atmosphere (Fig.10). As for **1**, the first step in the temperature range 27~155 °C corresponds to the loss of coordinated waters (Obsd. 3.21%, Calcd. 2.60%). The second step occurs at the range of 180~820 °C, corresponding to the loss of bpp and dtb ligands, and the remaining weight of 10.34% shows CoO component (Calcd. 10.71%). For **2**, the weight loss of 6.05% (Calcd. 5.38%) in the range of 25 to 250 °C reveals the exclusion of coordinated water molecules. The removal of organic ligands occurs from 190 °C to 910 °C and the remaining weight of 11.08% shows CoO component (Calcd. 11.19%).

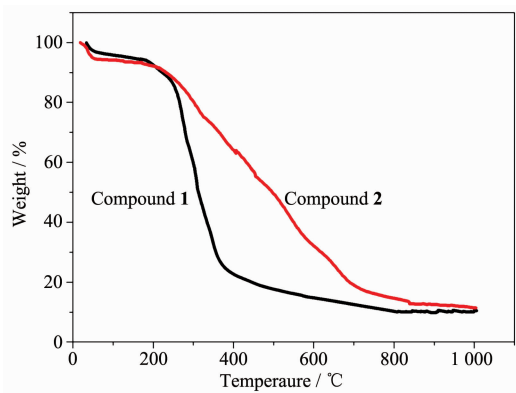


Fig.10 TGA curves of complexes **1** and **2**

3 Conclusions

In summary, the simultaneous use of H₂dtb and N-donor ligands to react with Co(II) atom affords two transition-metal polymeric framework. Complex **1** displays a 2D corrugated network containing a fascinating meso-helices structure. Compound **2** shows the same 1D chain which is formed a 2D supramolecular network by hydrogen bonding. The two neutral polymeric complexes exhibit structural and dimensional diversity due to the different coordination modes of the flexible dtb ligand and the effect of assistant ligands. Simultaneously, the V-shaped configuration of dtb ligand is considered as a good candidate for the construction of helix. Further investigations on such interesting systems connected by dtb ligand will be applied to other MOFs in our future work.

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