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Four coordination polymers derived from a one-pot reaction and their controlled synthesis

Wei Yan, Han Hao and Hegen Zheng*

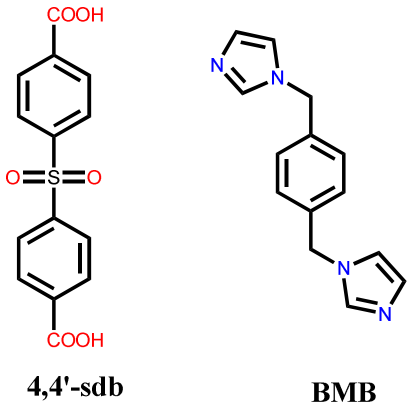
Abstract: Four different Co(II) coordination polymers have been built by two flexible ligands 4,4'-dicarboxydiphenyl sulfone (4,4'-sdb) and 1,4-bis((1*H*-imidazol-1-yl)methyl) benzene (BMB) in a one-pot solvothermal reaction. The structures of **1** and **2** are new, **3** and **4** have been reported. The crystal structures of **1-3** were obtained, namely, $[Co(4,4'\text{-sdb})(BMB)]_n$ (**1**); $\{[Co_2(4,4'\text{-sdb})_2(BMB)]\cdot 2H_2O\}_n$ (**2**), $[Co_3(4,4'\text{-sdb})_2(DMF)(H_2O)_3]_n$ (**3**), but **4** was confirmed by PXRD. Both **1** and **2** are 2D layered structure with **sql** topology and their point symbol is $\{4^4\cdot 6^2\}$. These complexes have been characterized by single crystal X-ray diffraction, infrared spectroscopy, thermogravimetry, elemental analysis, and powder X-ray diffraction measurements. By changing the synthesis conditions, four different Co(II) coordination polymers can be obtained respectively.

Introduction

The coordination polymers (CPs) have potential applications in luminescence,[1] molecular recognition,[2] magnet,[3] drug delivery,[4] heterogeneous catalysis,[5] gas storage,[6] sensing and separation[7] due to their diverse and fascinating structures. Certainly, the sructures of CPs were influenced by many factors such as metal centers, organic ligands, the ratio of reagents, temperature, reaction solvents, pH value, counterions.[8] Among those factors, the effect of solvent has been comfimed in lots of cases,[9-10] that different CPs could be selectively obtained using different solvents from the same components.

Two or more species with different architectures through cocrystallization has gained much attention in the last decades due to their structural diversity and great potential applications.[11] One-pot synthesis of two or three compounds has been investegated.[12] However, it is rare that four compounds are found in a one-pot reaction. Now, the biggest challenge is to obtain different kinds of one-component crystals by optimizing the experimental conditions. Different components have different structures. Great progress has not witnessed many comprehensive discussions on the control over topological structure, and the goal of predicting the exact structure and composition of coordination polymers remains a long-term project.[13]

In this paper, we use two flexible ligands, 1,4-bis ((1*H*-imidazol-1-yl)methyl) benzene (BMB) and 4,4'-dicarboxydiphenyl sulfone (4,4'-sdb) incorporating transition metal ions Co(II) to construct four coordination polymers from a one-pot reaction: $[Co(4,4'\text{-sdb})(BMB)]_n$ (**1**); $\{[Co_2(4,4'\text{-sdb})_2(BMB)]\cdot 2H_2O\}_n$ (**2**), $[Co_3(4,4'\text{-sdb})_2(DMF)(H_2O)_3]_n$ (**3**), $[(Co_2(BMB)_3]_n$ (4) (the formula was confirmed by PXRD and EA). Based on the significant differences of the first three structures, we adopt solvents to regulate, finally, four kinds of pure crystals have been obtained, respectively. All complexes are characterized by elemental analysis, infrared spectroscopy, powder X-ray diffraction, and thermogravimetry measurements. Furthermore, the crystal and topological structures, UV-Visible spectra are investigated in detail.



Scheme 1. Structures of the 4,4'-sdb and BMB ligands used in this work.

Results and discussion

The experimental section has been listed in the Supporting Information. The detailed information of complexes **1-2** is summarized in Table 1 and S1.

Description of the crystal structure of {[Co(4,4'-sdb)(BMB)]}$_n$ (1)

Complex **1** crystallizes in the orthorhombic space group *Cmca*. As shown in Fig. 1a, each Co(II) ion is coordinated by four carboxylate oxygen atoms from two different 4,4'-sdb ligands and two nitrogen atoms from two BMB ligands to adopt octahedral coordination modes. The Co-N bond lengths are 2.0521(18) Å, and Co-O bond lengths are 2.1527(18) and 2.1998(15) Å, which are all similar to those values found in other complexes.[14] 4,4'-sdb ligands adopt bidentate chelating mode to combine Co(II) ions to form 1D chains (Fig. 1b). Another *zig–zag* chains constructed by BMB ligands and Co(II) ions cross the above chains to form a 2D wave-like layer. When the parallel layers are stacking in an AAA···mode, infinite one-dimensional channels are created along the a-axis (Fig. 1c). The solvent-accessible volume of these channels, calculated with the PLATON program, is 1339.2 Å^3 or 21.1% of the total unit cell volume. From a topological perspective, the 2D layers can be simplified to 4-connected **sql** nets with point symbol $\{4^4 \cdot 6^2\}$.

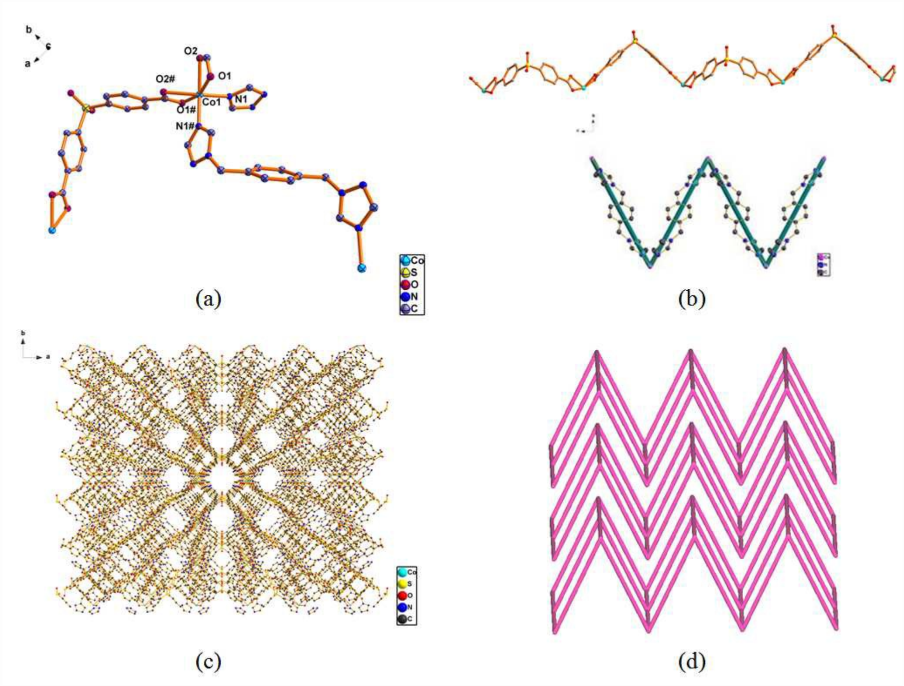


Fig. 1 (a) Coordination environment of complex **1**; (b) The 1D chain constructed by 4,4'-sdb ligands and Co(II) ions (up); the 1D chain constructed by BMB ligands and Co(II) ions (down); (c) One-dimensional channels along the *c*-axis; (d) Topological representation of the 2D layer of **1**.

Description of the crystal structure of $\{[Co_2(4,4'\text{-sdb})_2(BMB)]\cdot 2H_2O\}_n$ (2)

X-ray analysis reveals that complex **2** crystallizes in the triclinic space group $P\bar{1}$. The asymmetric unit contains one Co(II) ion, half a BMB ligand, one 4,4'-sdb ligand and two lattice H_2O molecules squeezed by PLATON software. As shown in Fig. 2a, each Co(II) is coordinated by four carboxylate oxygen atoms from four 4,4'-sdb ligands at the basal positions and two nitrogen atoms from two BMB ligands at the apical position. The Co-N bond length is 2.0337(15) Å, and the Co-O bond lengths are vary in the range of 2.0330(13)-2.1001(13) Å; the N-Co-O angles are in the range of 91.99(6)-94.28(6)°, and the O-Co-O angles are in the range of 86.65(5)-162.34(6)°. Two crystallographically equivalent Co(II) cations are bridged by four carboxylate groups adopting a bis-bidentate coordination mode to generate a dinuclear Co(II) “paddle-wheel” secondary building unit (SBU) with a Co···Co distance of 2.8706(4)

Å (Fig. 2a). The 4,4'-sdb ligands link the paddle-wheel to form a 1D chain. The BMB ligands link the “paddle-wheel” to form another 1D chain (Fig. 2b). Two chains intersected into a 2D flat network (Fig. 2c). The 2D layers are stacking in an AAA··· mode to form a one-dimensional channels (Fig. 2d). From the topological perspective, the SBU [$Co_2(CO_2)_4$] can be regarded as a 6-connected node; thus, the structure of **2** can be topologically represented as a 4-connected **sql** net with the vertex symbol of $\{4^4{\cdot}6^2\}$.

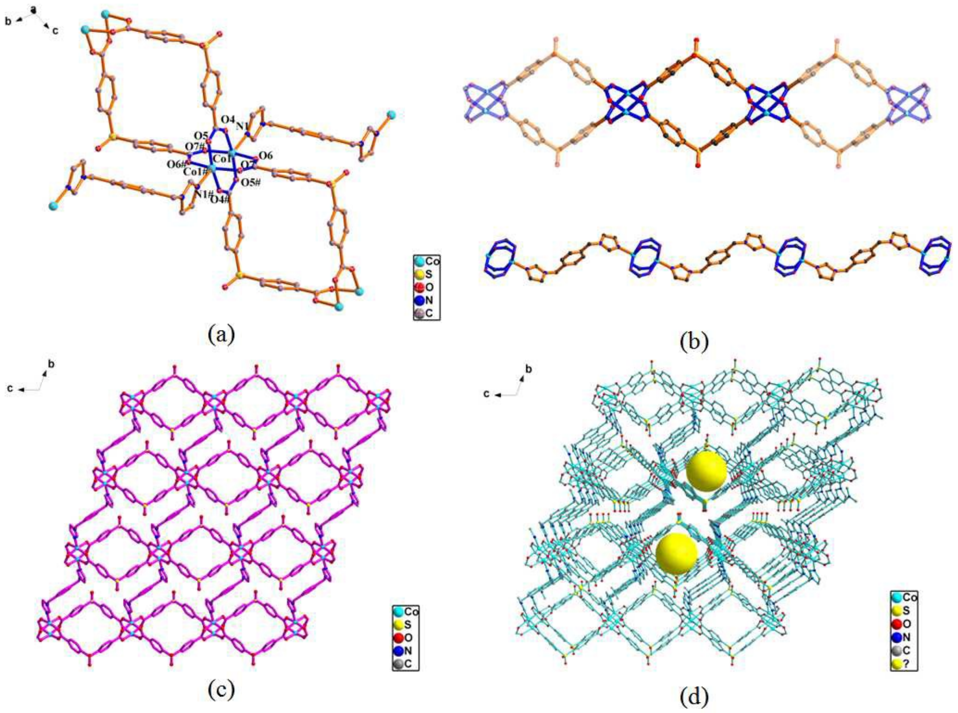


Fig. 2 (a) Coordination environment of complex **2**. The hydrogen atoms are omitted for clarity; (b) The chain linked by dinuclear Co(II) “paddle-wheel” SBU and 4,4'-sdb ligands (up); another chain formed by the “paddle-wheel” SBU and BMB ligands (down); (c) Two chains intersected into a 2D flat network; (d) The 2D layers are stacking in an AAA··· mode to form a one-dimensional channels.

Description of the crystal structures of $[Co_3(4,4'\text{-sdb})_2(DMF)(H_2O)_3]_n$ (3) and $[Co_2(BMB)_3]_n$ (4)

We are not going to discuss the structures of **3** and **4** in detail for they have been reported (The crystal samples of **4** suitable for the single crystal X-ray diffraction were not obtained, but its structure is the same with reference 20, we confirmed it through a lot of characterization such as PXRD, EA and so on).[15, 16] In complex **3**, carboxylate groups of 4,4'-sdb ligands adopting bidentate coordination mode to generate a rod-shaped SBU (Fig. 3a). This SBU extends along layer direction to form a 2D structure (Fig. 3b). As shown in Fig. 3c, each Co(II) ion in **4** is coordinated by six nitrogen atoms from six different BMB ligands and adopt octahedral coordination mode. The BMB ligands connected with Co(II) ions to form a 3D networks (Fig. 3d).

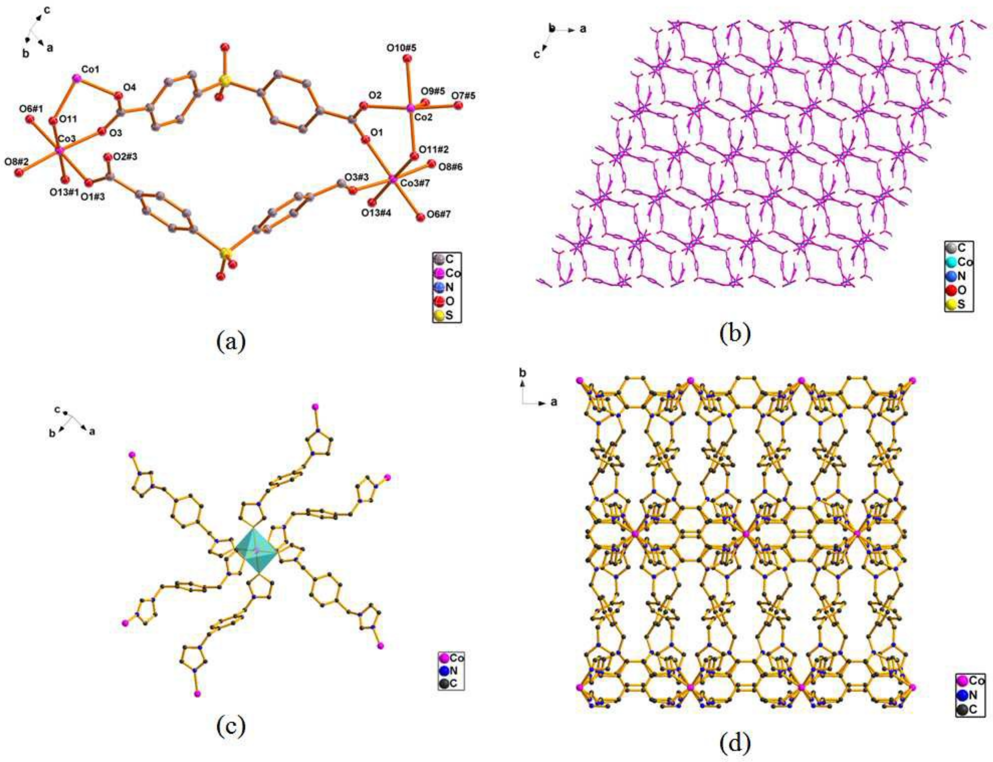


Fig. 3 (a) Coordination environment of **3**. The hydrogen atoms are omitted for clarity; (b) The 2D structure of **3**; (c) Coordination environment of **4**; (d)The 3D networks of **4**.

X-ray powder diffraction results

In order to check whether the crystal structures are truly representative of the bulk materials, the powder X-ray diffraction (PXRD) experiments for **1**–**4** were carried out at room temperature. As shown in Figs. S1–S4 (Supporting Information), the main peak positions of the simulated and experimental PXRD patterns are in agreement with each other.

Thermogravimetric analyses

In order to characterize the thermal stability of these complexes, thermogravimetric (TG) analyses (Fig. S5) were studied in detail. For complexes **1** and **4**, the structures can keep stable below 400 °C, appear high thermal stability. A weight loss of approximately 7.08% between 150-340 °C for **2** is equivalent of lossing two water molecules (calcd 6.95% for **2)**. From 390 °C, the skeleton of complex begins to collapse, which shows a rapid weight loss. For complex **3**, before 120 °C, the weightlessness is equivalent to the loss of water attached to the sample surface. Between 120 to 250 °C, the weight loss of 6.25% can be seen as the loss of coordinated DMF and H_2O molecules (calcd 6.34%). After 400 °C, the rapid weight loss can be regarded as the burning of the organic ligands, at the same time accompanied by the collapse of the skeleton.

UV-Visible spectra

The UV-visible spectra of ligands BMB, 4,4'-sdb and complexes **1-4** are presented in Fig. S12. The spectra of BMB and 4,4'-sdb ligands exhibit narrow absorption band in the range of 250–380 nm (absorption maxima is 320 nm for BMB and 295 nm for 4,4'-sdb, respectively), which can be ascribed to π–π* transitions of the ligands. Besides these two absorption bands, complex **1** also has wide absorption band at 400–670 nm and complex **2** at 415–625 nm. Compared with **1** and **2**, complexes **3** and **4** have relatively narrow absorption band at 427–583 nm and 453–653 nm. The lower energy bands can be considered as metal–to–ligand charge–transfer (MLCT) transitions.[17]

To explore the semiconductivity, the diffuse reflectivity is transformed into absorbance using the Kubelka–Munk function:[18]

$$F(R) = \frac{(1-R)^2}{2R} = \frac{K}{S}$$

with *R* representing the reflectance, *K* the absorption, and *S* the scattering. In a *K/S* vs

E (eV) plot, extrapolating the linear part of the rising curve to zero provides the onset of absorption. The band gap of complex **1** at 1.57 eV and 2.68 eV, while display a good semiconductor property (Fig. S12b). The value at 1.57 eV and 2.61 eV for complex **2**, 1.52 eV and 2.42 eV for **3**, 1.81 eV and 2.40 eV for **4**, respectively, which are estimated to be greater than the semiconductor silicon material with a band gap of 1.10 eV.

Optimal experiment for purifing one-component crystals of complexes 1-4.

Based on the significant differences of four structures, we adopt solvents to regulate, finally, three kinds of pure crystals have been obtained, respectively, we just obtained the power of **4** and this structure was confirmed by PXRD. The relative studies were conducted and observations are detailed in Table S2 (ESI): (1) when DMF/H_2O is used as solvent systems, the ratio changes from 1/5 (means 1 ml *vs.* 5 ml) to 5/1, we only obtain powder crystals. When H_2O is replaced by CH_3CN, only powder is obtained. Here, we have yet to obtain single crystals, and then we try to use the three-component solvent instead of two-component solvent systems. (3) When DMF/CH_3CN/H_2O is used as solvent systems, the ratio is 2/2/2, many multi-component crystals **1**-**4** were obtained. A few one-component crystals **1** were obtained in the ratio 3/0.5/2.5, crystals **2** were obtained in the ratio 1/1.5/3.5, crystals **3** were obtained in the ratio 2/0.5/3.5, the samples **4** was obtained in the ratio 1/0.5/4.5.

All above optimizational experiments were carried out with total amount of solvent of 6.0 ml at 95 °C for three days. From Table S2, plenty of powder was gained with the lack of CH_3CN or H_2O in solvent systems. However, clear solution will be generated if the solvent system is only DMF. For the most part, we obtained the components containing impurities or mixed components. This phenomenon demonstrated that selected solvent systems play a significant role in compound synthesis.

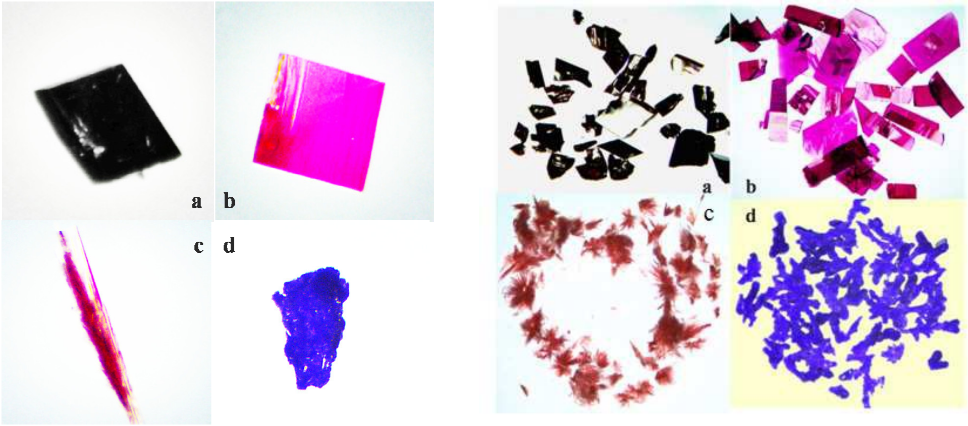


Fig. 4 The optical microscope photographs of complexes **1**-**4**.

Conclusions

In summary, four different Co-CPs with different structures have been co-crystallized in one-pot crystallization. From the proportion of four species in the initial products, through a serious of characterization, we try our best to optimize the synthesis conditions, finally, four kinds of one-component crystals have been obtained. Complexes **1** and **2** are both 2D motifs with a **sql** topology. Complex **3** is also a 2D structure constructed from rod-shaped SBUs. Complex **4** is a 3D networks, which was confirmed by PXRD, elemental analysis and so on.

Acknowledgment

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Notes and references

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Electronic Supplementary Information (ESI) available: Selected bond lengths and angles, patterns of photochemistry, TGA and PXRD; Crystallographic data in CIF format, CCDC: 1418239-1418240. See DOI: 10.1039/x0xx00000x.

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Table 1 Crystal data and structural refinements parameters of complexes **1-2**.

Complex	**1**	**2**
Empirical formula	$C_{26}H_{20}CoN_6O_6$ S	$C_{21}H_{19}CoN_2O_8S$
Formula weight	603.47	518.34
Crystal system	Orthorhombic	Triclinic
Space group	*Cmca*	$P\bar{1}$
a / Å	24.226(2)	7.0828(7)
b / Å	20.495(2)	12.9378(13)
c / Å	12.7686(12)	12.9943(13)
α / °	90	109.3550(10)
β/ °	90	92.9220(10)
γ/ °	90	101.7330(10)
V / Å^3	6339.8(10)	1090.90(19)
Z	8	2
D_{calcd} / g cm^{-3}	1.265	1.468
μ / mm^{-1}	0.652	0.922
F(000)	2472	492
θ min-max / °	1.68, 27.48	1.67, 27.64
Tot., uniq. data	20899, 3735	9991, 5001
R(int)	0.0314	0.0229
Nref, Npar	3735, 186	5001, 280
R_1, wR_2 [$I > 2\sigma(I)$]	0.0469, 0.1453	0.0322, 0.1131
GOF on F^2	1.093	1.035
Min. and max resd dens (e·Å^{-3})	-0.644, 1.097	-0.626, 0.594

$R_1=\Sigma||F_o|-|F_c||/|\Sigma|F_o|$; $wR_2=\{\Sigma[w(F_o^2-F_c^2)^2]/\Sigma[w(F_o^2)^2]\}^{1/2}$; where $w=1/[\sigma^2(F_o^2)+(aP)^2+bP]$,$P=(F_o^2+2F_c^2)/3$.

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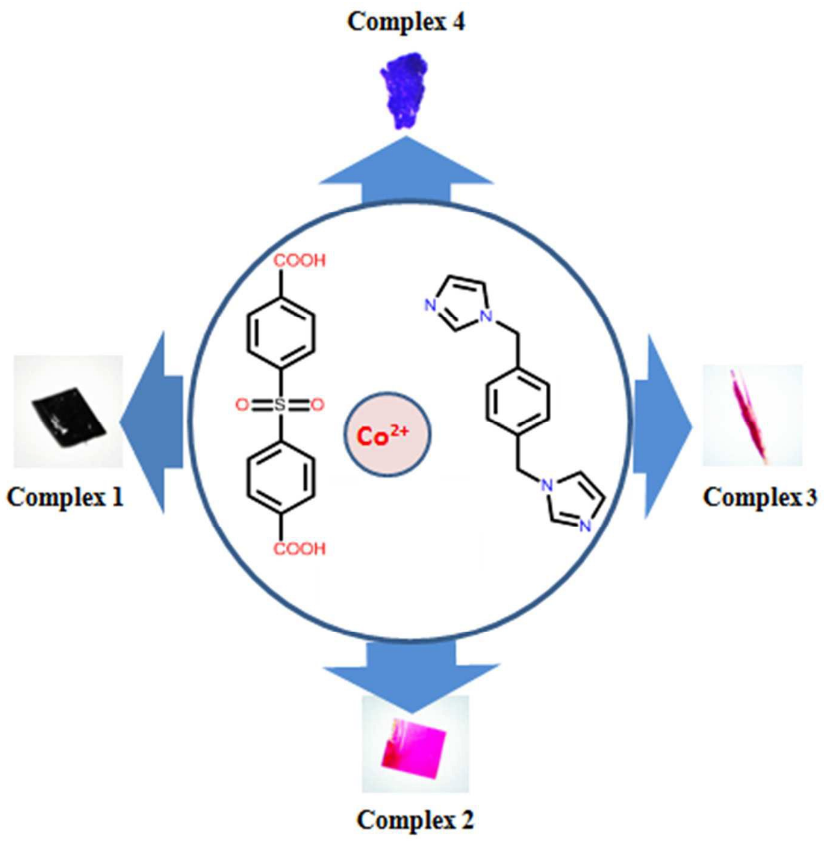

Four different Co(II) coordination polymers have been built by two flexible ligands in a one-pot solvothermal reaction. All the complexes show optical semiconductive properties.