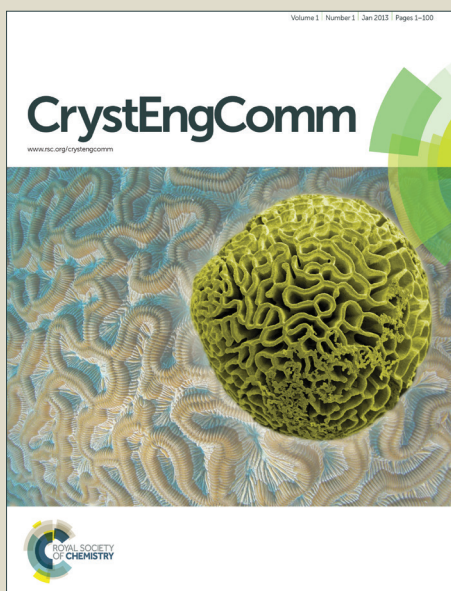


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Two 2D silver complexes from symmetric to noncentrosymmetric architecture controlled by metal-ligand ratio

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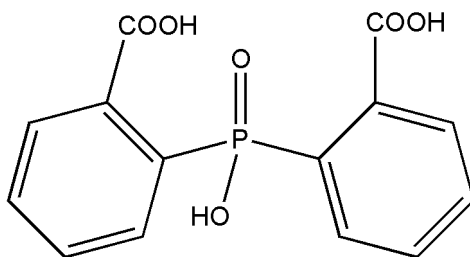
The hydrothermal reaction of silver nitrate and 2,2'-phosphinico-dibenzoic acid (H₃L) with different metal to ligand ratio under hydrothermal condition yielded two unusual complexes with the formulae of [Ag(H₂L)]_n (**1**) and [Ag₃(L)]_n (**2**), respectively. Both complexes were well characterized by single-crystal X-ray diffraction, elemental analysis, IR spectroscopic, and fluorescence studies. Complex **1** crystallizes in a centrosymmetric *Pbcn* space group and presents a two-dimensional (2D) layer structure through silver(I)-aromatic interaction, whereas complex **2** adopts a noncentrosymmetric *Iba2* space group, in which different coordination modes of the four groups around the phosphorus atom in L³⁻ ligand occurs. Both complexes display green emissions, assigned as ligand-to-metal charge-transfer character.

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Introduction

Coordination polymers formed by linking metal ions through multifunctional ligands are attracting wide attention due to their great potential in gas adsorption, catalysis, ion exchange, molecular magnets, photoluminescence as well as their beautiful architecture.¹ The two primary factors in controlling the structural features of these coordination polymers are the organic linkers and the coordination geometry adopted by the metal ions.² However, their structures are also subjected to many other subtle influences, such as solvent, pH and metal-ligand ratio.³ It has been reported that altering the metal to ligand ratio in one reaction system can produce various coordination polymers with different dimensions and/or symmetries under certain circumstances.⁴ For example, Carlucci prepared two silver complexes from 2D layer to 3D network by careful control of the molar ratio between silver ions and the hexamethylenetetramine ligand.^{4a} Zhang synthesized a family of mixed valence Mn coordination clusters, ranging from high symmetric $\{\text{Mn}^{\text{II}}\text{Mn}^{\text{III}}_{12}\}$ to noncentrosymmetric $\{\text{Mn}^{\text{II}}_3\text{Mn}^{\text{III}}_{10}\}$ and than chiral $\{\text{Mn}^{\text{II}}_3\text{Mn}^{\text{III}}_{11}\}$ core, by adjusting the concentrations of Cl^- .^{4b} Nevertheless, it is still a great challenge to obtain multi-dimensional noncentrosymmetric complexes from symmetrical moieties by varying the metal to ligand ratio. Fortunately, employing silver nitrate and 2,2'-phosphinico-dibenzoic acid (H_3L) (**Scheme 1**) as two reactants, we obtained two 2D silver complexes from symmetric to noncentrosymmetric architecture by the adjustment of the metal to ligand ratio. Herein, we report the syntheses, crystal structures and luminescent properties of the two complexes $[\text{Ag}(\text{H}_2\text{L})]_n$ (**1**) and $[\text{Ag}_3(\text{L})]_n$ (**2**) in detail.



Scheme 1. Chemical Structure of Ligand H_3L .

Experimental Section

Materials and Physical Measurements. All reagents and solvents used in the present work were of analytical grade as obtained from commercial sources without further purification. The ligand 2,2'-phosphinico-dibenzoic acid (H_3L) was synthesized according to the literature.⁵

Elemental analyses for C, H were performed on a Vario EL- III elemental analyzer. The FT-IR spectra were recorded using KBr pellets in the range from 4000 to 400 cm^{-1} on a Nicolet Avatar A370 spectrophotometer. Powder X-ray diffraction (PXRD) data for **1** and **2** were collected on a DX-2700 diffractometer with Cu $K\alpha$ radiation ($\lambda = 1.5406 \text{ \AA}$) over the 2θ range of 5 - 30° at room temperature. Luminescence spectra of the solid samples were recorded on a Shimadzu RF-5310 spectrophotometer.

Synthesis of $[Ag(H_2L)]_n$ (1**).** $AgNO_3$ (51 mg, 0.3 mmol), H_3L (30.6 mg, 0.1 mmol) were sealed in a 15 ml Teflon-lined stainless steel autoclave with 6 mL H_2O and heated at 120 °C for 3 days. After cooling to room temperature at a rate of 10 °C \cdot h⁻¹, colorless prism-shaped crystals of **1** were obtained in 68% yield based on the H_3L ligand. Elemental analysis calcd (%) for $C_{14}H_{10}O_6PAg$ (413.07): C, 40.67; H, 2.42. Found: C, 40.91; H, 2.52. IR / cm^{-1} (KBr): 3445(m), 3094(w), 2740(m), 2356(m), 1887(m), 1674(s), 1583(m), 1555(m), 1470(m), 1415(m), 1297(s), 1271(s), 1127(s), 1085(s), 1014(s), 816(m), 799(s), 767(s), 731(s), 699(m), 649(m), 558(s).

Synthesis of $[Ag_3(L)]_n$ (2**).** Complex **2** was obtained by a similar procedure of **1** except that the molar ratio of $AgNO_3$ (153 mg, 0.9 mmol) and H_3L (30.6 mg, 0.1 mmol) was 9:1. Colorless rod-like crystals were isolated by filtration and washed by water. The yield is 87% based on H_3L . Elemental analysis calcd (%) for $C_{14}H_8O_6PAg_3$ (626.78): C, 26.80; H, 1.28. Found: C, 25.54; H, 1.33. IR / cm^{-1} (KBr): 3442(m), 3086(w), 3052(w), 1545(s), 1514(s), 1469(w), 1434(w), 1395(s), 1380(s), 1263(w), 1180(w), 1151(m), 1136(m), 1113(s), 1076(m), 1006(m), 959(w), 876(w), 837(m), 785(m), 754(s), 727(m), 708(m), 657(m), 579(m), 560(s).

X-ray Crystallography. Single-crystal X-ray diffraction data for complexes **1** and **2** were collected on a Bruker SMART APEX CCD diffractometer using graphite monochromatized Mo $K\alpha$ radiation ($\lambda = 0.71073 \text{ \AA}$) at room temperature using the *phi* and *omega* scan technique. Data reduction was conducted with the Bruker SAINT package. Absorption correction was performed

using the SADABS program. The structure was solved by direct methods and refined on F^2 by full-matrix least-squares using SHELXL-2000 with anisotropic displacement parameters for all non-hydrogen atoms. H atoms were introduced in calculations using the riding model. The crystallographic data and structural refinement results are summarized in Table 1. The selected bond distances and angles are listed in Table 2. CCDC 980093 and 980094 contain the details.

Table 1 Crystal data and structure refinement information for **1** and **2**.

Complexes	1	2
Formula	C ₁₄ H ₁₀ O ₆ PAg	C ₁₄ H ₈ O ₆ PAg ₃
Formula weight	413.06	626.78
Crystal system	Orthorhombic	Orthorhombic
Space group	<i>Pbcn</i>	<i>Iba2</i>
<i>a</i> (Å)	7.9663(9)	18.151(3)
<i>b</i> (Å)	11.4723(13)	26.476(4)
<i>c</i> (Å)	14.0805(17)	5.9595(10)
α (°)	90	90
β (°)	90	90
γ (°)	90	90
<i>V</i> (Å ³)	1286.8(3)	2864.0(8)
<i>Z</i>	4	8
<i>D_c</i> (g cm ⁻³)	2.132	2.907
<i>F</i> (000)	816	2368
GOF (F^2)	1.145	1.050
Flack parameter	—	0.00(3)
<i>R</i> ₁ ^a	0.0276,	0.0194,
<i>wR</i> ₂ ^b [<i>I</i> > 2σ(<i>I</i>)]	0.0749	0.0549

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

Table 2 Selected bond distances (Å) and angles (°) for **1** and **2**.

Complex 1			
Ag1-O3	2.527(3)	Ag1-O3 ^a	2.527(3)
Ag1-C5 ^b	2.568(3)	Ag1-C5 ^c	2.568(3)
O3-Ag1-O3 ^a	58.80(11)	O3 ^a -Ag1-C5 ^c	128.84(9)
O3-Ag1-C5 ^b	128.84(9)	C5 ^b -Ag1-C5 ^c	116.16(15)
O3 ^a -Ag1-C5 ^b	107.10(9)	O3-Ag1-C5 ^c	107.10(10)
Complex 2			
Ag1-O5 ^a	2.276(3)	Ag1-O2	2.332(3)
Ag1-O6 ^b	2.451(3)	Ag2-O6 ^a	2.347(3)
Ag2-O3	2.420(3)	Ag2-O1	2.472(3)
Ag2-O1 ^c	2.550(3)	Ag2-O2 ^d	2.597(3)
Ag3-O4	2.464(3)	Ag3-O4 ^a	2.229(4)
Ag3-O3 ^c	2.297(3)		
O5 ^a -Ag1-O2	135.81(13)	O5 ^a -Ag1-O6 ^b	94.62(13)
O2-Ag1-O6 ^b	119.24(10)	O1 ^c -Ag2-O2 ^d	82.58(10)
O6 ^a -Ag2-O3	144.81(10)	O6 ^a -Ag2-O1	115.40(11)
O3-Ag2-O1	87.08(10)	O6 ^a -Ag2-O1 ^c	85.66(10)
O3-Ag2-O1 ^c	126.53(11)	O1-Ag2-O1 ^c	79.92(6)
O6 ^a -Ag2-O2 ^d	77.95(11)	O3-Ag2-O2 ^d	91.35(10)
O1-Ag2-O2 ^d	156.89(10)	O4 ^a -Ag3-O3 ^e	139.76(12)
O4 ^a -Ag3-O4	120.79(12)	O3 ^e -Ag3-O4	88.83(12)

Symmetry codes: a: $-x+1, y, -z+1/2$; b: $-x+1/2, y-1/2, z$; c: $x+1/2, y-1/2, -z+1/2$; d: $x-1/2, y+1/2, z+1/2$. (2)
a: $-x+1/2, -y+1/2, z-1/2$; b: $x-1/2, -y+1/2, z$; c: $-x, y, z-1/2$; d: $x, y, z-1$; e: $-x+1/2, -y+1/2, z+1/2$; f: $-x+0, y+0, z+1/2$; g: $x, y, z+1$; h: $x+1/2, -y+1/2, z$.

Results and discussion

Preparations

Both complexes **1** and **2** were obtained by hydrothermal reactions of AgNO_3 and ligand H_3L . In complex **1**, only the phosphinico group of ligand H_3L is deprotonated (*vide infra*), whereas both the phosphinico and the carboxyl groups are deprotonated in complex **2** (*vide infra*). Therefore, we conclude that the ligand H_3L was deprotonated in a stepwise fashion as the increase of the metal-ligand ratio. To obtain complexes in pure form, the optimization of the experimental procedure was conducted by varying the metal to ligand ratio systematically. The powder X-ray diffraction (PXRD) patterns of the products at different metal-ligand ratio, including 2:1, 4:1, 6:1, 8:1, and 10:1, were displayed in Fig. 1. As can be seen, a mixture of complexes **1** and **2** was harvested when the metal-ligand ratio were ranging from 4:1 to 8:1. Therefore, metal-ligand ratio of 3:1 and 9:1 were selected respectively for the synthesis of **1** and **2**.

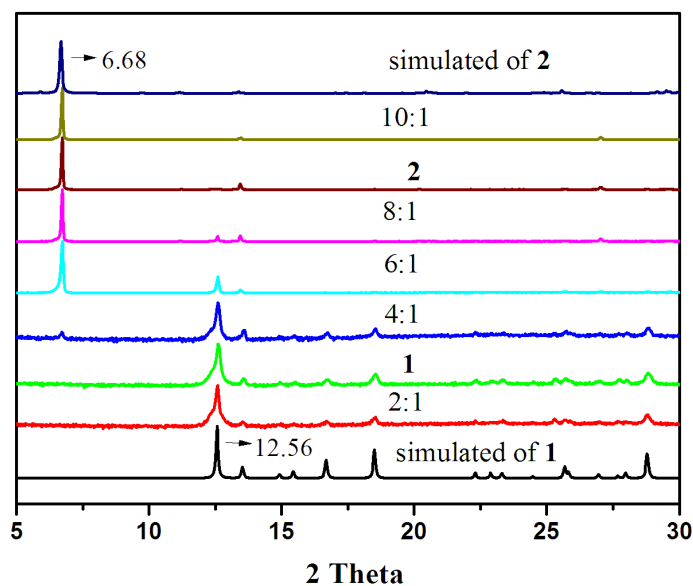


Fig.1 PXRD patterns of complexes **1**, **2** and products with different metal-ligand ratio.

Crystal structures

[Ag(H₂L)]_n (1). The single crystal X-ray analysis reveals that complex **1** crystallizes in the orthorhombic space group *Pbcn*, and shapes into a 2D sheet. The asymmetric unit contains half of a H₂L⁻ ligand and half of a Ag(I) ion. As shown in Fig. 2a, the silver metal center is chelated by two phosphinico oxygen atoms from one H₂L⁻ ligand and two phenyl carbon atoms from another two different H₂L⁻ ligands. According to the description of Houser,⁶ the Ag ions adopts a distorted trigonal pyramidal coordination geometry ($\tau_4 = 0.72$) with two larger θ angles of 128.85(9)°. The Ag–O and Ag–C bond distances are 2.527(3) and 2.568(3) Å, respectively, consistent with other Ag(I) complexes reported in the literature.⁷ In complex **1**, the H₂L⁻ anion lies on the inversion center and each connects three Ag(I) ions (Fig. 2b), extending to a 2D layer in the *ab* plane (Fig. 3).

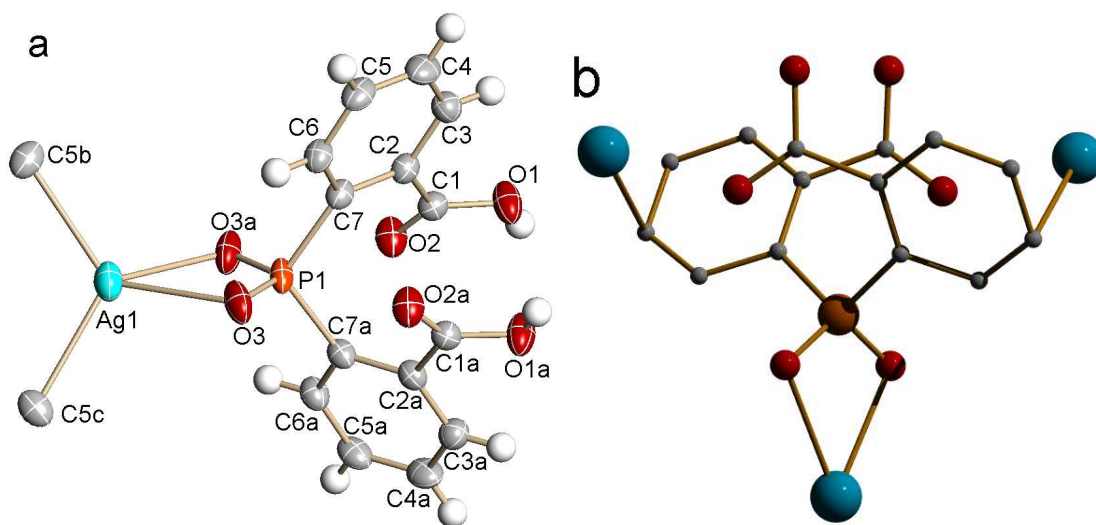


Fig. 2 Molecular structure of **1** with 50% probability displacement ellipsoids. Symmetry code: a: $-x+1, y, -z+1/2$; b: $-x+1/2, y-1/2, z$; c: $x+1/2, y-1/2, -z+1/2$.

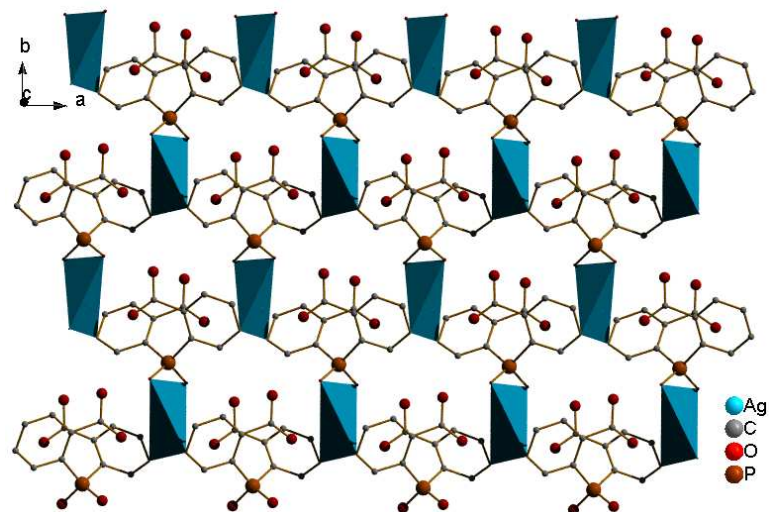


Fig. 3 2D layer in the *ab* plane of complex 1.

[Ag₃(L)]_n (2). Increasing the metal-ligand ratio, complex 2 crystallizes in the space group of *Iba2*, and is an extended 2D network. As depicted in Fig. 4a, the asymmetric unit contains one L³⁻ anion and three crystallographically independent Ag(I) ions. Both Ag1 and Ag3 adopt trigonal planar geometry. The Ag1 ion is coordinated to three carboxylate oxygen atoms from three L³⁻ ligands, whereas Ag3 is bound to three phosphinico oxygen atoms from three L³⁻ ligands. The two AgO₃ groups are significantly distorted from planar with the O–Ag–O angles of 135.81(13), 119.24(10) and 94.62(13)^o for Ag1, 139.76(12), 120.79(12) and 88.83(12)^o for Ag3. Therefore, the two Ag ions are located 0.42 and 0.41 Å out of the plane defined by the three oxygen atoms coordinated to the metal atoms. Different from the aforementioned two Ag ions, the Ag2 is in a distorted trigonal bipyramidal configuration. The Ag–O distances are in the range of 2.23 - 2.60 Å (Table 2), falling in the typical range of Ag–O bond.^{7a} Each L³⁻ anion connects ten Ag(I) ions with its six oxygen atoms. It is notable that, although the L³⁻ anion has a symmetrical structure, the connection modes for the carboxylate groups and phosphinico group are obviously different (Fig. 4b). The first carboxylate group (O1-C1-O2) links four Ag(I) ions through a bridging mode. The other one (O5-C14-O6) links three Ag(I) ions through a monodenate O5 atom and a bridging O6 atom. The O3 and O4 atoms of phosphinico group, each bridges two Ag(I) ions, while O1 atom from the same ligand is coordinated to the Ag2 atom. Thus the four groups around the phosphorus atom in the L³⁻ ligand connect ten Ag(I) ions

through different coordination modes to form noncentrosymmetric complex **2**, which crystallizes in a space group *Iba*2 with a Flack parameter 0.00(3).⁸

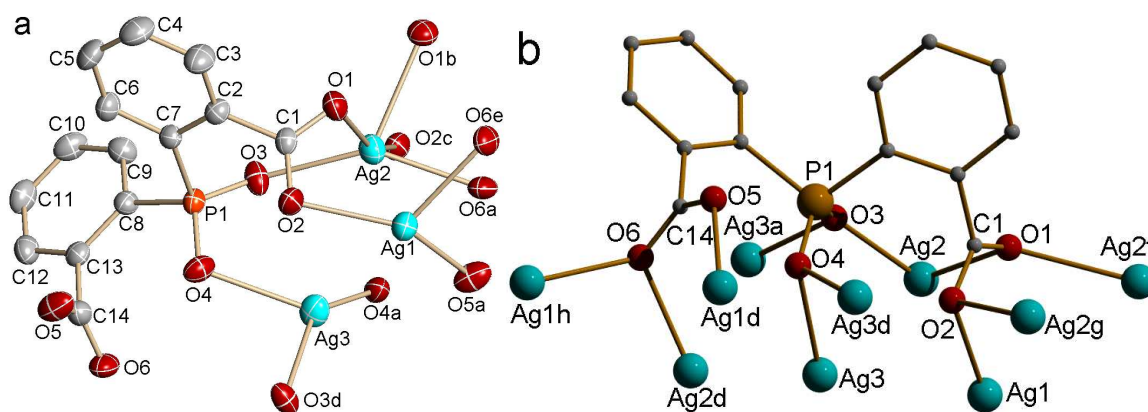


Fig. 4 (a) Molecular structure of **2** with 50% probability displacement ellipsoids. (b) Coordination modes of the four groups around the phosphorus atom in L^{3-} ligand. Hydrogen atoms are omitted for clarity. Symmetry code: a: $-x+1/2, -y+1/2, z-1/2$; b: $-x, y, z-1/2$; c: $x, y, z-1$; d: $-x+1/2, -y+1/2, z+1/2$; e: $x-1/2, -y+1/2, z$; f: $-x+0, y+0, z+1/2$; g: $x, y, z+1$; h: $x+1/2, -y+1/2, z$.

The Ag(I) ions are interconnected by bridging L^{3-} anions into a complicated 2D hybrid layer, which is parallel to the *ac* plane. The interconnection of the Ag(I) ions by bridging oxygen atoms of L^{3-} anions form the main skeleton of the layer. In the skeleton, Ag3 are bridged by O4 to form a zig-zag chain, whereas Ag1 and Ag2 are linked by the carboxylate oxygen atoms to form a ladder chain. The two chains are further interconnected by O3 with alternately arrangement (Fig. 5a), leading to moderate Ag \cdots Ag interactions [3.1610(7) – 3.2983(7) Å].⁹ The overall structure can be described as layers stacking along the *c* axis, with the phenyl groups oriented toward the interlayer space (Fig. 5b).

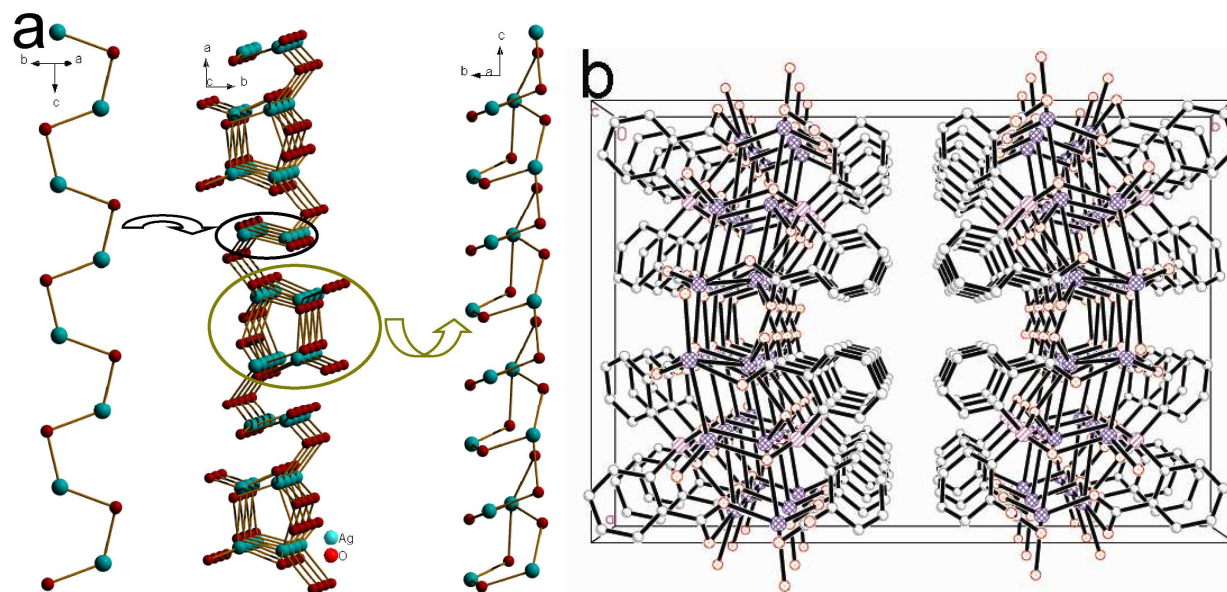


Fig. 5 The main skeleton formed (a) and the packing structure of **2** along c axis (b). Hydrogen atoms are omitted for clarity.

IR and luminescent studies

Infrared spectra of **1** and **2** show strong characteristic bands of P=O in the region of 1395 to 1014 cm^{-1} and middle bands of P–O around 1050–850 cm^{-1} .¹⁰ For complex **1**, a strong peak at 1674 cm^{-1} indicates the existence of the –COOH group. In addition, the absence of absorption bands in the range of 1600–1500 cm^{-1} proves that the two carboxyl groups maintain protonated, which is in agreement with the crystallographic studies of **1**. In the IR spectrum of **2**, there is not the characteristic band of –COOH near 1700 cm^{-1} , but we observed the characteristic strong absorption bands in 1545–1514 cm^{-1} and 1395–1380 cm^{-1} which are assigned to the asymmetric and symmetric stretching vibrations of carboxylate groups respectively.¹¹

The luminescent spectra of complexes **1** and **2** as well as the free ligand H₃L were measured in the solid state at room temperature (Fig. 6). The free ligand H₃L displays emission at 350 nm upon the irradiation of 317 nm light, which is probably attributed to $n \rightarrow \pi^*$ or $\pi \rightarrow \pi^*$ transition. The emissions of **1** and **2** are observed at 508 nm ($\lambda_{\text{ex}}=320$ nm) and 496 nm ($\lambda_{\text{ex}}=328$ nm), respectively, which are significantly different from that of free ligand. Compared to the emission of the free ligand, the luminescence of **1** and **2** can be assigned to ligand-to-metal charge transfer (LMCT).¹² Since the luminescence of d^{10} systems has a demonstrated sensitivity to metal–metal

interactions, the existence of Ag(I)⋯Ag(I) interactions is expected to play a role in the solid state emission, which are likely responsible for the low energies of the luminescence bands of **2**.¹³

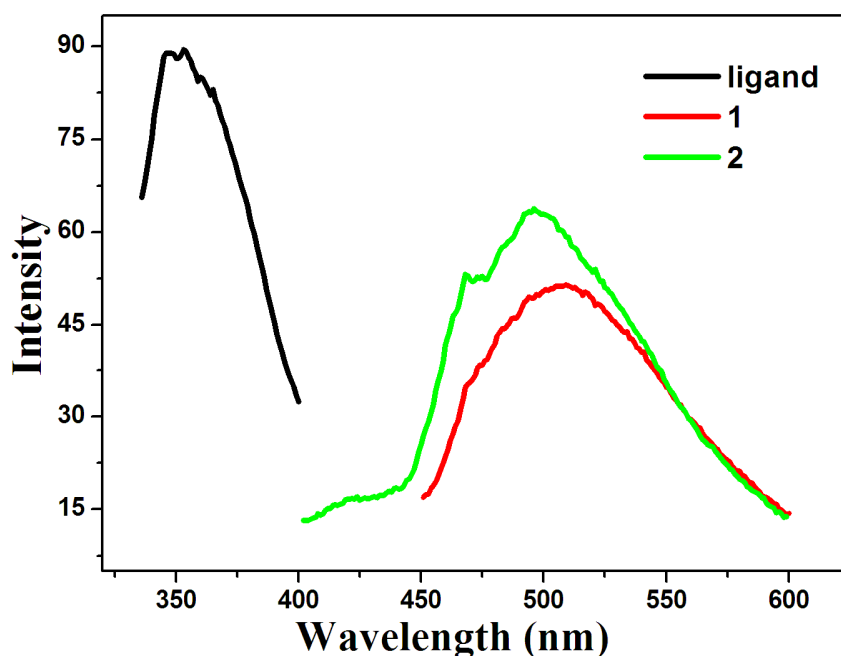


Fig. 6 The solid-state photoluminescent spectra of ligand, complexes **1** and **2**.

Conclusions

In summary, two 2D silver(I) coordination polymers have been successfully synthesized by adjusting the metal-ligand ratio under hydrothermal reaction and they are also structurally characterized by X-ray diffraction analyses. Through the strong silver(I)-aromatic interaction, complex **1** extends into a 2D layer. Increasing the metal-ligand ratio leads to the formation of complex **2**, a noncentrosymmetric 2D architecture is constructed by different connections of the four groups around the phosphorus atom. This work also offers a rational synthetic strategy to obtain multi-dimensional noncentrosymmetric complexes based on symmetrical materials by controlling the metal-ligand ratio.

Associated Content

Supporting Information.

X-ray crystallographic files (CIF) of complexes **1** and **2**.

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