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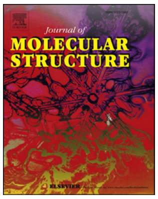

Solvent-controlled assembly of crystal structures: From centrosymmetric structure to noncentrosymmetric structure

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ABSTRACT

Reported here are two isomeric organic crystals and two HgI_2-based coordination compounds by solvo(hydro)thermal method: $[(TPTA)\cdot H_2O]_n$ (**1**, *Cc*), $[(TPTA)\cdot H_2O]_n$ (**2**, *Pbca*), $[Hg_{1.5}I_3(TPTA)_{0.5}(CH_3CN)\cdot(H_2O)_{0.5}]_n$ (**3**, *P2₁*) and $[HgI_2(TPTA)\cdot H_2O]_n$ (**4**, *P2₁/c*) (TPTA = N,N′,N″-tris(3-pyridyl) trimesic amide). Single crystal X-ray diffraction show that they afford noncentrosymmetric and centrosymmetric structures, respectively. Note that this kind of formations can be precisely controlled by changing the reaction solvent, thus indicating a facile method towards generating noncentrosymmetric structure.

1. Introduction

Synthesis and design of coordination compounds (CCs) have attracted ever increasing interest due to not only their structural novelty but also promising applications in the fields of adsorptions, catalysis, luminescence, magnetism, conductivity, nonlinear optics (NLO), etc [1–4]. However, the formation of the CCs is highly influenced by various reaction conditions, such as solvent system, reaction temperature, pH value of the solution, ratio of ligand to metal ion, and coordination geometry of central metals and organic ligands [5–8].

In particular, the coordination compounds (CCs) crystals with noncentrosymmetric structures (NCS) have received significant attention, owing to the extensive potential applications in ferroelectric, pyroelectric, piezoelectric, and second harmonic generation (SHG) properties [9–11]. Furthermore, to date, it remains a considerable challenge to control the structures to NCS [12,13]. Generally, noncentrosymmetric materials can be well-designed by using preferred coordination geometries of metal centers and/or carefully chosen bridging ligands [14]. Several methods have been developed to synthesize coordination compounds with noncentrosymmetric structures. First of all, it is to use optically pure chiral multidentate ligands, which has been proved to be the most effective and straightforward approach [15,16]. Then, we can use of asymmetric ligand or flexible ligand to prepare noncentrosymmetric materials [17]. Moreover, noncentrosymmetric compounds can be also obtained through unpredictable spontaneous resolution and topology-building [18,19]. The investigation of spontaneous resolution has been carried out by many researchers during the past decades. It has been found that spontaneous resolution occurs in the course of crystallization is closely related to many external factors which are similar as to the formation of CCs [20–22]. Therefore, it is such a challenging work to accurately control the crystals from centrosymmetric structure to noncentrosymmetric structure by changing the reaction conditions. Herein, based on a tripodal ligand of N,N′,N″-tris(3-pyridyl) trimesic amide (TPTA), we found that the presence of a small quantity of water molecules as co-solvents would control the final structures from centrosymmetry to noncentrosymmetry.

2. Experimental

2.1. Materials and methods

Commercially available reagents were used throughout without further purification and TPTA was synthesized according to the literature method [23–26]. Elemental analyses for C, H, and N were performed with a Perkin–Elmer 2400 Series II element analyzer. Thermogravimetric analysis (TGA) experiments were carried out with a DuPont Thermal Analyzer from room temperature to 800 °C

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Table 1
Selected bond lengths/Å and bond angles/° for **3** and **4**.

Compound **3**			
Hg(1)–N(2)	2.376(7)	N(2)–Hg(1)–I(3)	104.09(17)
Hg(1)–I(3)	2.6321(7)	N(2)–Hg(1)–I(4)	102.08(17)
Hg(1)–I(4)	2.6650(7)	I(3) –Hg(1) –I(4)	149.87(3)
Hg(1)–I(4B)	3.1741(7)	N(2)–Hg(1)–I(4B)	90.47(18)
Hg(2)–N(1)	2.413(6)	I(3)–Hg(1)–I(4B)	102.66(2)
Hg(2)–I (1)	2.6347(7)	I(4)–Hg(1)–I(4B)	91.74(2)
Hg(2)–I(2)	2.6341(7)	N(1)–Hg(2)–I(1)	99.23(19)
Hg(2)–I(1A)	3.3248(7)	N(1) –Hg(2) –I(1A)	97.9(2)
Hg(3)–N(3)	2.392(7)	I(2)–Hg(2)–I(1)	158.80(2)
Hg(3)–I(5)	2.6731(7)	I(2)–Hg(2)–I(1A)	93.79(2)
Hg(3)–I(5B)	3.5271(7)	I(1)–Hg(2)–I(1A)	89.01(2)
Hg(3)–I(6)	2.6305(7)	N(3)–Hg(3)–I(6)	102.64(16)
Hg(3)–I(6A)	3.3960(7)	N(3) –Hg(3) –I(5)	100.99(17)
		I(6)–Hg(3)–I(5)	156.36(3)
		Hg(1)–I(4)–Hg(1A)	91.74(2)
		Hg(2)–I(1)–Hg(2B)	89.01(2)
Compound **4**			
Hg(1)–N(1)	2.396(6)	N(1)–Hg(1)–N(6A)	88.5(2)
Hg(1)–N(6A)	2.441(6)	N(1)–Hg(1)–I(1)	104.17(16)
Hg(1)–I(1)	2.6290(9)	N(6)–Hg(1)–I(1A)	103.01(15)
Hg(1)–I(2)	2.6581(9)	N(1)–Hg(1)–N(2)	106.43(16)
Hg(1)–N(6B)	2.441(6)	N(6)–Hg(1)–I(2A)	100.97(16)
		I(1)–Hg(1)–I(2)	141.26(3)

Symmetry code: (A): 1 + x, y, z; (B): −1+x, y, z.

in a nitrogen atmosphere at a heating rate of 10 °C min^{-1}. In the measurements of emission and excitation spectra, the pass width is 5.0 nm.

2.2. Synthesis of [(TPTA)·H_2O]$_n$ (**1**)

N,N′,N″-tris(3-pyridyl) trimesic amide(TPTA) (0.2 mmol) with 5 mL DMF and 1 mL H_2O was mixed in a Teflon container and sealed in an autoclave. The autoclave was heated to 115 °C in a programmable oven, and this temperature was maintained for three days before it was cooled to room temperature. Light yellow block-shaped crystals were obtained. EA analysis (%): calc. C/63.15, N/18.41, H/4.42; exp. C/63.17, N/18.48, H/4.40.

2.3. Synthesis of [(TPTA)·H_2O]$_n$ (**2**)

N,N′,N″-tris(3-pyridyl) trimesic amide(TPTA) (0.2 mmol) with 5 mL DMF was mixed in a Teflon container and sealed in an autoclave. The autoclave was heated to 115 °C in a programmable oven, and this temperature was maintained for three days before it was cooled to room temperature. Light yellow bar-type crystals were obtained. EA analysis (%): calc. C/63.15, N/18.41, H/4.42; exp. C/63.14, N/18.42, H/4.45.

2.4. Synthesis of [$Hg_{1.5}I_3(TPTA)_{0.5}(CH_3CN)\cdot(H_2O)_{0.5}$]$_n$ (**3**)

The mixture of HgI_2 (0.2 mmol) and TPTA(0.2 mmol) in the 5 ml acetonitrile and 1 mL H_2O solution was stirred for 30 min, and then transferred to a Teflon container and sealed in an autoclave. The autoclave was heated to 160 °C in a programmable oven, and this temperature was maintained for three days before it was cooled to room temperature (cooling rate 3°C/h). Light yellow crystals were obtained in the yield of 65% based on Hg. EA analysis (%): calc. C/17.70, N/5.90, H/1.27; exp. C/17.73, N/5.90, H/1.26.

2.5. Synthesis of [$HgI_2(TPTA)\cdot H_2O$]$_n$ (**4**)

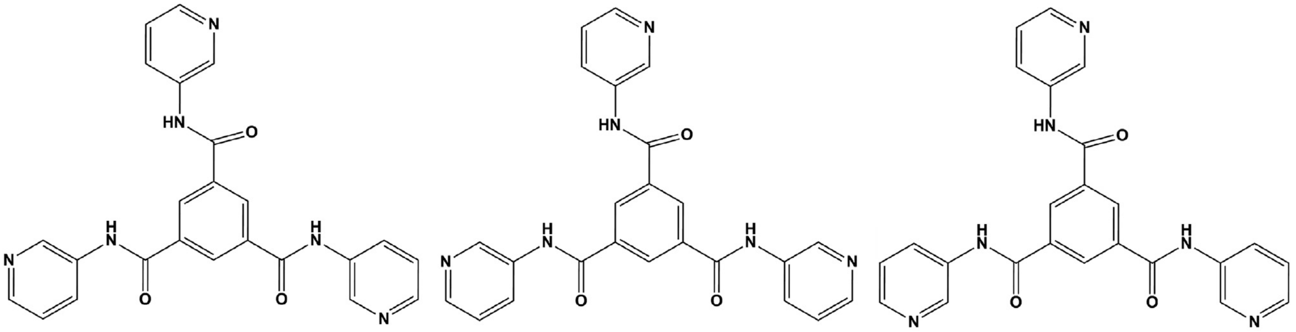
The mixture of HgI_2 (0.2 mmol) and TPTA(0.2 mmol) in the 5 ml acetonitrile solution was stirred for 30 min, and then transferred to a Teflon container and sealed in an autoclave. The autoclave was heated to 160 °C in a programmable oven, and this temperature was maintained for three days before it was cooled to room temperature (cooling rate 3°C/h). Light brown crystals were obtained in the yield of 68% based on Hg. EA analysis (%): calc. C/32.29, N/9.41,H/2.03; exp. C/32.27, N/9.45, H/2.01.

2.6. Crystallographic measurements

Single-crystal diffraction intensity data were collected on Bruker Smart Breeze CCD employing graphite-monochromated Mo-*Kα* radiation ($\lambda = 0.71073$ Å) at 296(2)K, the measurement method is phi and omega scans, and corrected with SADABS program. The structures were solved by direct methods and all non-hydrogen atoms were subjected to anisotropic refinement by full matrix least-squares on F^2 using SHELXTS 97 and SHELXTL 97 program [29,30]. Non-hydrogen atoms were refined anisotropically and hydrogen atoms were placed in calculated positions and refined isotropically using a riding model. The important interatomic distances and angles are listed in Table 1, and The crystallographic data

Table 2
Crystal data, experimental conditions, and structure refinement parameters of **1**, **2**, **3** and **4**.

	1	**2**	**3**	**4**
Empirical formula	$C_{24}H_{20}O_5N_6$	$C_{24}H_{20}N_6\ O_3$	$C_{14}H_{12}Hg_{1.5}I_3N_4O_2$	$C_{24}H_{18}Hg\ I_2N_6O_3$
Formula weight/g mol	456.46	456.46	949.86	892.83
Temperature/K	296(2)	296(2)	296(2)	296(2)
Wavelength/Å	0.71073	0.71073	0.71073	0.71073
Crystal size/mm	0.30 × 0.28 × 0.25	0.20 × 0.20 × 0.18	0.20 × 0.20 × 0.19	0.20 × 0.20 × 0.18
Crystal system	Monoclinic	Orthorhombic	Monoclinic	Monoclinic
Space group	*Cc*	*Pbca*	$P2_1$	$P2_1/c$
D_{calc}/g cm^{-3}	1.423	1.404	2.934	2.369
a/Å	17.7919(14)	8.1083(15)	4.2062(3)	14.429(5)
b/Å	14.5737(9)	22.087(4)	29.6215(19)	19.984(7)
c/Å	8.3397(7)	24.109(4)	17.2646(10)	8.724(3)
β/°	99.900(5)	90.00	91.269(3)	95.681(9)
V/Å^3	2130.2(3)	4317.6(13)	2150.5(2)	2503.2(15)
Z	4	8	4	4
F(000)	952	1904	1676	1656
θ range for data collection/°	1.82–25.00°	1.69–25.00°	1.37–25.00°	1.75–25.00°
Reflections collected	8049	16981	15040	16463
R_{int}	0.0429	0.0627	0.0513	0.0457
GooF	1.079	1.071	1.111	1.100
Final *R* indices $[I < 2\sigma(I)]$ [a]	$R_1 = 0.0456$, $wR_2 = 0.0845$	$R_1 = 0.0595$, $wR_2 = 0.1252$	$R_1 = 0.0646$, $wR_2 = 0.1573$	$R_1 = 0.0395$, $wR_2 = 0.1036$
R indices (all data) [b]	$R_1 = 0.0587$, $wR_2 = 0.0948$	$R_1 = 0.0982$, $wR_2 = 0.1442$	$R_1 = 0.0668$, $wR_2 = 0.1599$	$R_1 = 0.0430$, $wR_2 = 0.1070$
Largest diff. peak and hole/e Å^{-3}	0.128, −0.158	0.203, −0.209	4.112, −2.160	1.431, −1.655



trans-cis-cis *trans-trans-cis* *cis-cis-cis*

Scheme 1. The conformations of the ligand in crystal structures (*trans* and *cis* are defined based on the relative orientation of the amide oxygen and the pyridyl nitrogen. *trans*-conformation means the two atoms are in the opposite side. *cis*-conformation means that the pyridine nitrogen atom and the amide oxygen atom of the ligand are in the same side).

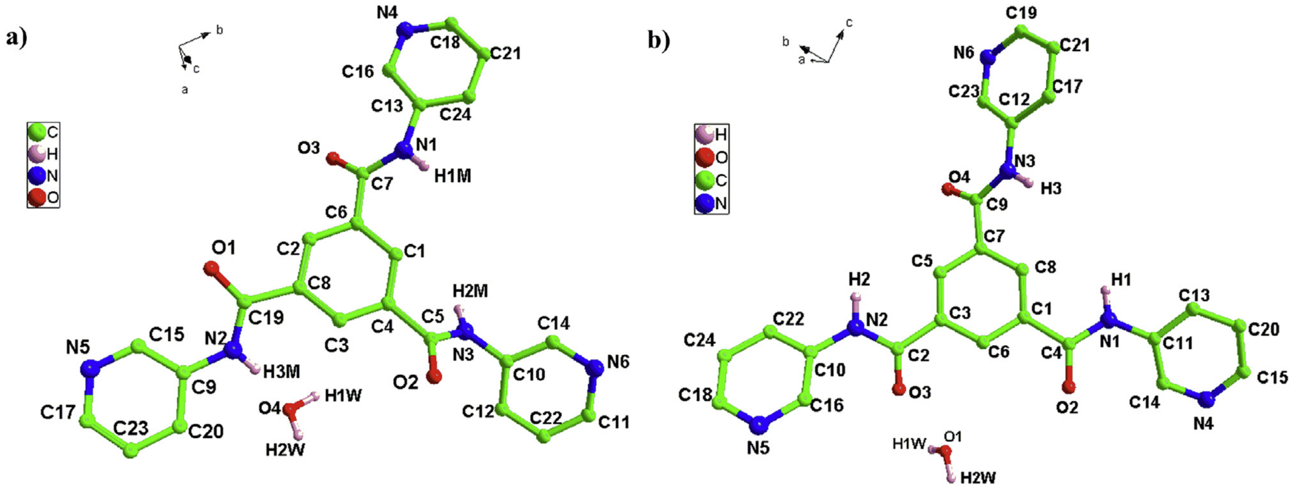


Fig. 1. View of the molecular structure of L for the asymmetric unit in **1** (a) and **2** (b).

for compounds **1**, **2, 3** and **4** are listed in Table 2.

3. Results and discussion

Organic molecular crystals **1** and **2** are isomers. **1** was synthesized with N,N′,N″-tris(3-pyridyl) trimesic amide(TPTA) in DMF-water(5 mL/1 mL) solution at 115 °C. The preparation of **2** is similar to that of **1** except that the solvent mixture was replaced with DMF (5 mL). Single crystal X-ray diffraction reveals that **1** crystallizes in the monoclinic acentric *Cc* space group with Flack factor −0.201(12), while **2** crystallizes in the orthorhombic space group *Pbca*. The coordination compound **3** was synthesized with HgI_2 and TPTA ligand in acetonitrile-water(5 mL/1 mL) solution by solvo(hydro)thermal reaction at 160 °C, while the coordination compound **4** was obtained under the same synthetic conditions but in acetonitrile (5 mL).

Single crystal X-ray diffraction reveals that compound **3** crystallizes in the acentric $P2_1$ monoclinic space group with Flack factor of 0.1, whereas compound **4** crystallizes in the $P2_1/c$ monoclinic space group. X-ray diffraction reveals that the C_3-symmetric triangular ligand of TPTA in **1**, **2**, **3**, **4** perform these conformations as follows: *cis-cis-trans*, *cis-cis-cis*, *trans-trans-cis*, *cis-cis-cis* (Scheme 1), where *cis*-conformation means that the pyridine nitrogen atom and the amide oxygen atom of the ligand are in the same side, and *trans*-conformation means that the two atoms are in the opposite side. Then, a query on Cambridge Crystallographic Data Centre (CCDC) also discloses that TPTA ligand in the *cis-cis-trans*-conformation and *trans-trans-cis*-conformation usually results in acentric coordination compounds, by contrast, TPTA ligand in the *cis-cis-cis*-conformation and *trans-trans-trans*-conformation often generate centrosymmetric coordination compounds. This is well consistent with our results, indicating that acentric or centrosymmetric feature of TPTA or TPTA coordination compounds is highly relative to the conformation of TPTA ligands.

3.1. Crystal structures

The asymmetric unit of both **1** and **2** containing one N,N′,N″-tris(3-pyridyl) trimesic amide (TPTA) molecule and one isolated water molecule are shown in Fig. 1. The terminal pyridyl groups of **1** are not co-planar with the central phenyl ring plane with dihedral angles of ca. 8.76°, 27.40°, 9.45°(Fig. 1a), while in **2** the corresponding values are ca. 37.47°, 25.23°, 20.23°, indicating more intense distortion of the TPTA ligand in **2** (Fig. 1b). TPTA molecules in **1** create a two-dimensional (2D) layer along *c* axis through N–H···O (N–H···O = 2.773(2), 2.786(2), 3.061(2) Å/161.40(4), 160.62(4), 160.85(4)°) and O–H···N (O–H···N = 2.847(3), 2.869(3) Å/174.37(4), 169.80(4)°) hydrogen bonds between the amide groups (Fig. 2a, c). While in **2**, it also create a two-dimensional (2D) layer but V-shaped along *a* direction through N–H···O (N–H···O = 2.996(2), 2.998(2) Å/150.69(4), 159.46(4)°) and O–H···N (O–H···N = 2.767(3), 2.942(3) Å/165.77(4), 170.59(4)°)

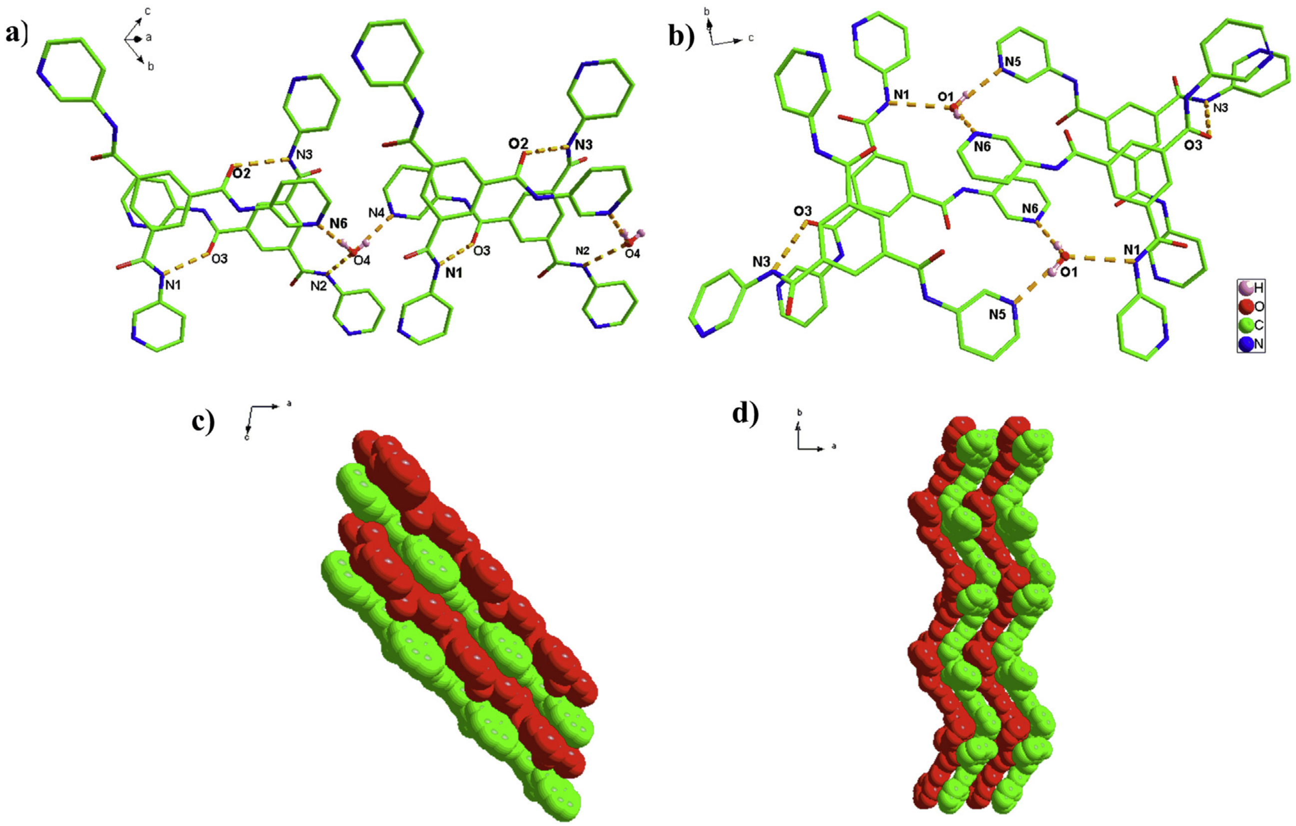


Fig. 2. (a) The hydrogen bonds of compound **1** are represented by yellow dashed lines. (b) The hydrogen bonds of compound **2** are represented by yellow dashed lines. (c) Layered-stacking structure of compound **1** along *c* direction. (d) V-shaped layer stacking structure of compound **2** along *a* direction. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

hydrogen bonds between the amide groups (Fig. 2b, d).

As for compound **3**, TPTA ligand in the *trans-trans-cis*-conformation acts as a trigonal bridge to combine three Hg(II) centers together. The asymmetric unit contains three unique Hg(II) ions(Fig. 3). Both Hg1, Hg2 show the four-coordinated tetrahedral geometry but Hg3 shows the five-coordinated pyramidal geometry

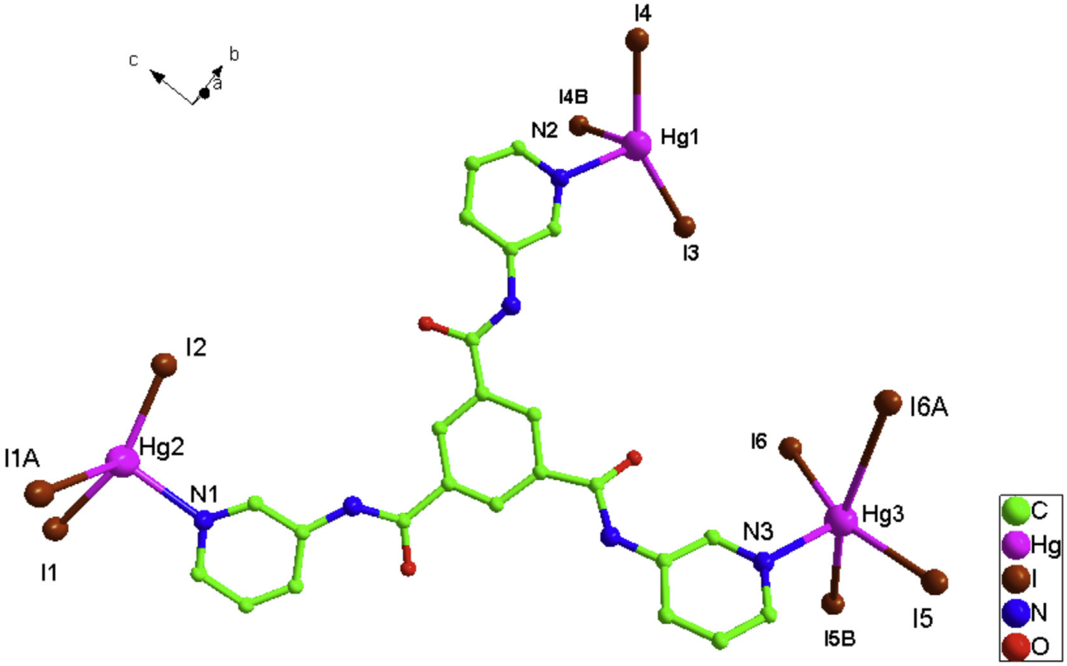


Fig. 3. The coordination surrounding around metal ions Hg(II) in **3**. Symmetry transformations used to generate equivalent atoms: A: 1 + x, y, z; B: −1+x, y, z. All hydrogen atoms are omitted for clarity.

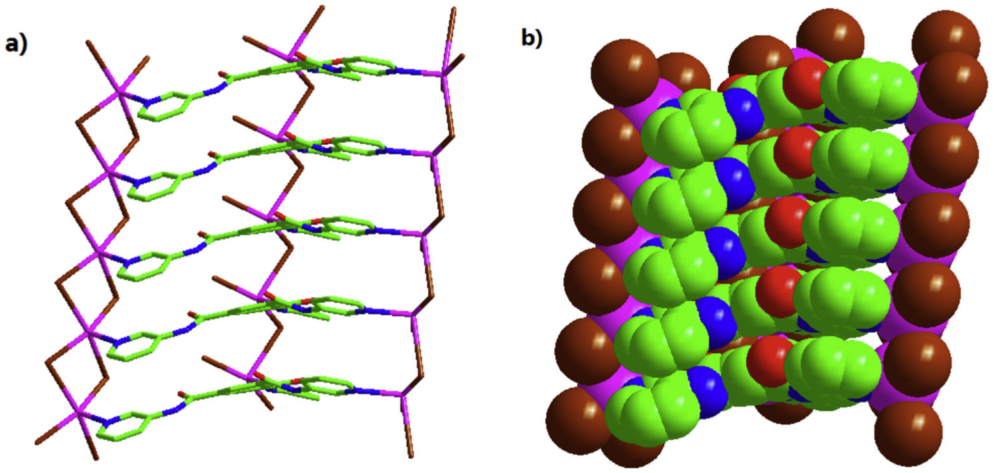


Fig. 4. (a) The one-dimensional columnar pillar with sideboard-like channel of compound **3**; (b) The one-dimensional columnar pillar stacking structure of compound **3**.

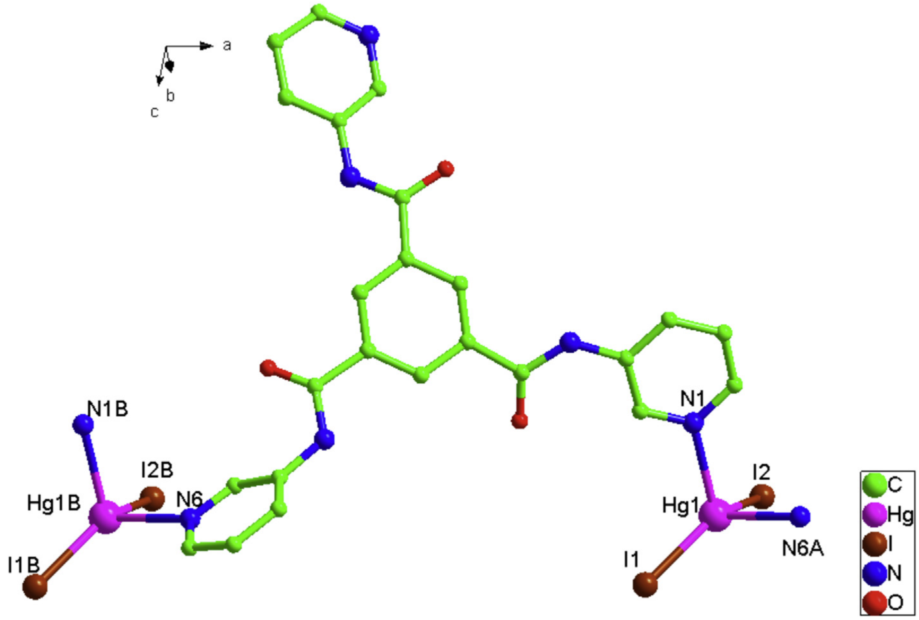


Fig. 5. The coordination surrounding around metal ions Hg(II) in **4**. Symmetry transformations used to generate equivalent atoms: A: 1 + x, y, z; B: −1+x, y, z. All hydrogen atoms are omitted for clarity.

and the coordination surrounding of them is described in detail below. Hg1 is coordinated by N2 from TPTA ligand and I3, I4, I4B (B: −1+x, y, z) from iodine ions I$^-$; Hg2 is coordinated by N1 from TPTA ligand and I1, I2, I1A (A: 1 + x, y, z) from iodine ions I$^-$; Hg3 is coordinated by N3 from TPTA ligand and I5, I5B, I6, I6A from iodine ions I$^-$. The lengths of Hg–I bond range from 2.631 Å to 3.527 Å,

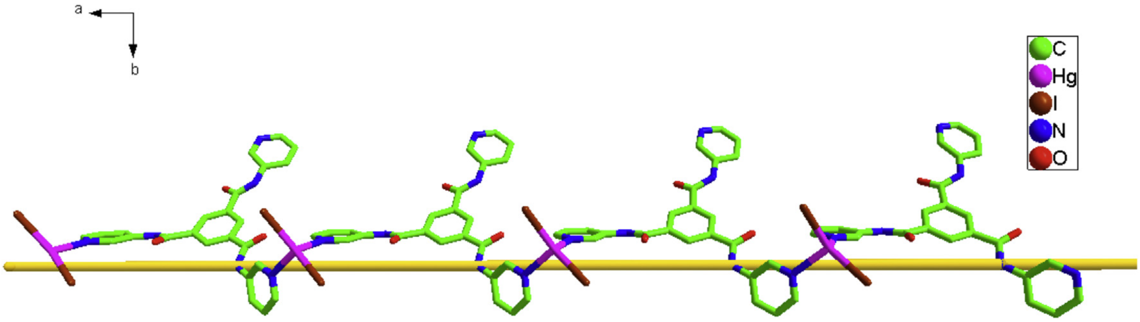


Fig. 6. View of the one-dimensional chain structure of compound 4 (yellow stick represented screw axis). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

which are all less than the sum of Hg (2.3 Å) and I (1.98 Å) van der Waals radius. And the bond lengths of Hg1–N2, Hg2–N1 and Hg3–N3 are 2.376(7)Å, 2.413(6)Å and 2.392(7)Å. The three terminal pyridyl groups of TPTA are not co-planar with the central benzene ring plane, giving the dihedral angles of ca. 20.16°, 28.27°, 20.24°.

Compound [$HgI_2(TPTA)\cdot H_2O]_n$ (**4**) presents different structure from **3**, and the coordination surrounding drawing is shown in Fig. 5, the asymmetric unit of **4** only contains one crystallography-independent Hg(II) ion, which displays the four-coordinated HgI_2N_2 tetrahedral geometry completed by two iodine atoms (I1, I2) and two TPTA nitrogen atoms (N1, N6). The bond lengths of Hg1–I1 and Hg1–I2 are 2.6290(9)Å, 2.6581(9)Å, respectively, while the bond lengths of Hg1–N1 and Hg1–N2 are 2.396(6)Å, 2.6581(9) Å. For TPTA ligand in the *cis-cis-cis* conformation, it takes the bridging mode to connect with two Hg(II) ions with one pyridyl nitrogen atom uncoordinated, and the corresponding dihedral angles are ca. 13.58°, 59.38°, 34.49°, also indicating more intense distortion of the TPTA ligand in **4**. As shown in Fig. 4a, the Hg(II) ions of **3** are integrated together to create a 1D columnar pillar with sideboard-like channel (Fig. 4a, b). Nevertheless, in **4**, along a axis direction, the Hg(II) ions are integrated together to create a 1D chain with a screw axis (Fig. 6).

3.2. TG-DTA, and photoluminescence properties

The thermostability of **3** and **4** are explored by TG studies at 35–800 °C (Fig. 7). Thermogravimetric analysis of **3** reveals that the weight loss (exp. 10.0%) in the temperature range of 35–200 °C is derived from one water molecule and two acetonitrile molecule (calc. 10.5%). For **4**, the first weight loss, 1.8%, at 35–150 °C is ascribed to the loss of one guest water molecules (calc. 2.0%). And then the chemical decomposition temperature is estimated around 300 °C. Compared with **3** and **4**, we can obviously see that compound **4** has higher thermal stability than **3**.

The solid-state photoluminescence properties of **3** and **4** were measured. As shown in Fig. 8, an intense emission of **3** occurs at 444 nm with an excitation wavelength of 313 nm, and **4** also displays one distinct emission peak at 441 nm under an excitation wavelength of 322 nm. As we know, the emission is mainly derived from the $\pi^*\rightarrow n$ or $\pi^*\rightarrow\pi$ transition. Meanwhile, the free TPTA ligand exhibits a 435 nm blue emission under 320 nm excitation [27], which indicates that the emissions of **3** and **4** originate from the TPTA ligand, and the red shift (9 nm, 6 nm) is mainly due to a metal-to-ligand or ligand to-metal charge transfer [28].

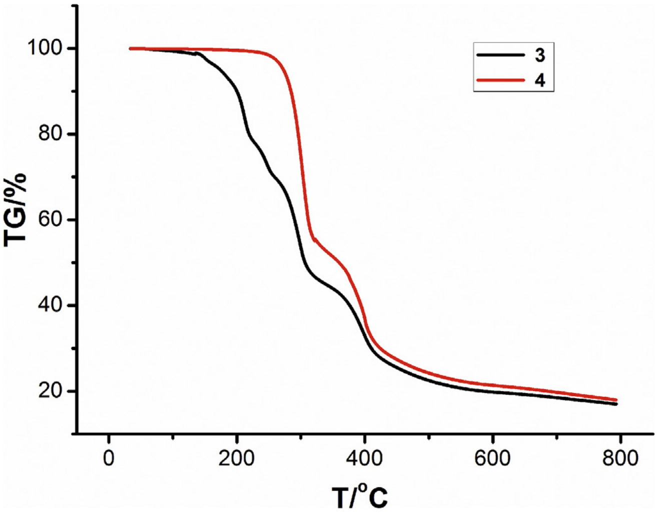


Fig. 7. The thermostability of **3** compound and compound **4**.

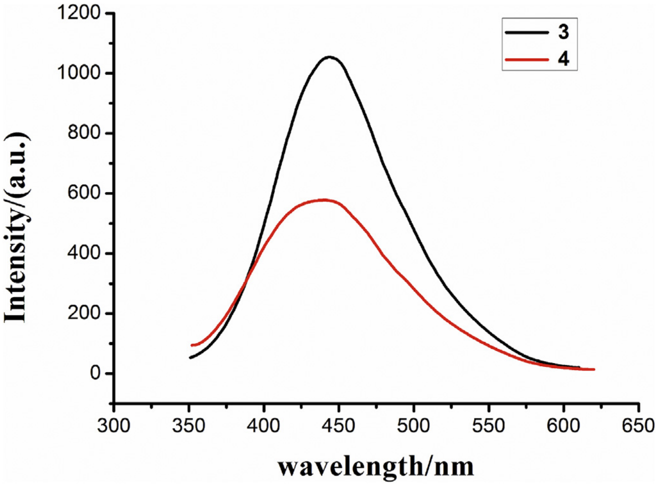


Fig. 8. The solid-state emission spectrum of compound **3** and compound **4**.

4. Conclusion

In conclusion, we successfully synthesized two isomeric organic crystals and two HgI_2-based coordination compounds using C_3-symmetric triangular ligand TPTA by solvo(hydro)thermal method. We through a good deal of the literature researches find that the compound structures are strongly correlated with the conformation of C_3-symmetric triangular ligand. In the synthesis of compound **1** and **3**, we used some water as co-solvents to obtain two noncentrosymmetric structures with TPTA ligands in *cis-cis-trans*-conformation or *trans-trans-cis*-conformation. Importantly, based on a tripodal ligand of N,N′,N″-tris(3-pyridyl) trimesic amide (TPTA), we found that the presence of a small quantity of water molecules as co-solvents would control the final structures from centrosymmetry to noncentrosymmetry. To some extent, the results have opened a facile method towards generating noncentrosymmetric structure.

Acknowledgment

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Appendix A. Supplementary materials

Crystallographic data (excluding structure factors) for the structures in this paper have been deposited with the Cambridge Crystallographic Data Centre, CCDC, 12 Union Road, Cambridge CB21EZ, UK. Copies of the data can be obtained free of charge on quoting the depository numbers CCDC-1061831 (**1**), CCDC-1061832 (**2**), CCDC-1061833 (**3**) and CCDC-1061831 (**4**) (Fax: +44-1223-336-033; E-Mail: deposit@ccdc.cam.ac.uk, http://www.ccdc.cam.ac.uk).

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