(+)-*N*-对甲苯磺酰-*L*-谷氨酸和 1,3-联(4-吡啶基)丙烷构筑的银(I)配位聚合物的合成、晶体结构及性质研究

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摘要:以手性配体(+)-*N*-对甲苯磺酰-*L*-谷氨酸(H_2tsgluO)和 1,3-联(4-吡啶基)丙烷(bpp)与银盐反应合成了一种同手性的配合物 $\{Ag(HtsgluO)(bpp)\}_n$ (**1**),对其进行了表征。X-射线单晶衍射测定表明,该配合物属单斜晶系,$P2_1$ 空间群,a=1.220 79(18) nm,b=1.034 49(16) nm,c=2.059 5(3) nm,β=96.006(2)°,V=2.586 7(7) nm^3,Z=4,R_1=0.060 2,银离子处于扭曲的四面体配位环境中,分别和 1 个 HtsgluO$^-$氧原子,2 个 bpp 氮原子以及 1 个银离子配位。配合物通过银银键构成了二维层状结构,并且进一步通过氢键构成了三维超分子结构。此外,对配合物的热重和荧光性质进行了研究。

关键词:银(I)配合物;(+)-*N*-对甲苯磺酰-*L*-谷氨酸;晶体结构
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Synthesis, Crystal Structure and Properties of a Silver(I) Complex Constructed from (+)-*N*-Tosyl-*L*-glutamic Acid and 1,3-Bis(4-pyridyl)propane

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Abstract: A homochiral complex $[Ag(HtsgluO)(bpp)]_n$ (**1**) was synthesized and characterized by the reaction of chiral (+)-*N*-Tosyl-*L*-glutamic acid (H_2tsgluO) and 1,3-bis(4-pyridyl)propane (bpp) with silver salts. Single crystal X-ray diffraction analysis reveals that complex **1** crystallizes in the monoclinic space group $P2_1$, a=1.220 79(18) nm, b=1.034 49(16) nm, c=2.059 5(3) nm, β=96.006(2)°, V=2.586 7(7) nm^3, Z=4, and final R=0.060 2. The silver atom lies in a distorted tetrahedral environment formed by one HtsgluO$^-$ oxygen, two bpp nitrogen and one symmetry-related silver(I) ions. It exhibits an infinite two-dimensional (2D) layered structure assembled by the argentophilic Ag-Ag interactions. Furthermore, these 2D layers are extended through extensive hydrogen bonding interactions to form an infinite 3D supramolecular network. In addition, the properties of thermogravimetric analysis and photoluminescent behavior of the complex have also been discussed. CCDC: 795083.

Key words: silver(I) complex; (+)-*N*-Tosyl-*L*-glutamic acid; crystal structure

0 Introduction

The synthesis and characterization of coordination polymers has been extensively studied over the past few decades because of their intriguing structures and, more importantly, their potential applications as functional materials[1-11]. A particularly interesting and challenging area in this field is the synthesis of homochiral metal-organic frameworks (MOFs) and exploration of their potential applications in heterogeneous

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asymmetric catalysis, enantioselective separations, chiral recognition, and second-order nonlinear optical (NLO)materials[12-19]. However, control the chirality in MOFs is still one of the main challenges. It is known that homochiral coordination polymers can be achieved by two general synthetic strategies based on the ligands used: one is using chiral organic ligand as linker to connect metal centers or metal clusters[12,20-26], and the another one is employing achiral ligand via spontaneous resolution on crystallization without any chiral auxiliary, which is relatively rare and will result in a racemic mixture of enantiomeric crystals[27-32]. It is believed that the usage of chiral species as reactant precursors represent the more straightforward and effective approach toward homochiral solids for it is much easier to control the chirality of the aimed compounds by taking advantage of the chirality of organic species. The selection of amino acid and their derivatives as chiral building units in constructing homochiral coordination polymers is attracting more and more attention in recent years due to their remarkable structural features and physical properties[21-22,33-40].

Extending our previous work on the self-assembly of coordination polymers based on (+)-*N*-Tosyl-*L*-glutamic acid (H_2tsgluO), an enantiomerically pure derivative of glutamic acid, and bidentate aromatic *N*-donor bases[41-42], we have explored the design and synthesis of homochiral coordination polymers employing chiral H_2tsgluO ligand and the flexible 1,3-bis (4-pyridyl)propane ligand (bpp), which can assume different conformations (TT, TG, GG and GG′;T=trans, G=gauche) with respect to relative orientations of CH_2 groups[43-44]. It is noteworthy that both ligands H_2tsgluO and bpp are flexible because flexible ligands can have variable conformation and coordination modes to satisfy the needs for the formation of ultimate chiral compounds.

We report here the synthesis, structural characterization, thermogravimetric analysis and the luminescent property of a monovalent silver metal bpp-bearing H_2tsgluO coordination polymer, wherein the ligand H_2tsgluO is monodeprotonated and exhibits a novel coordination mode that has not been reported so far. To the best of our knowledge, it represents the first example of silver(I) complex employing chiral H_2tsgluO and bridging heterocylic *N*-donor ligand.

1 Experimental

1.1 Materials and measurements

All reagents were of analytical grade and were used as obtained from commercial sources without further purification. Elemental analyses (C, H, N) was performed with an Elementar Vario EL elemental analyzer. IR (KBr pellets) spectra was recorded in the 400~4 000 cm^{-1} range with a FTIR-8900 spectrometer. Thermogravimetric analyses (TGA) were performed on a TGA-7 instrument in an N_2 atmosphere with a heating rate of 15 ℃·min^{-1}. Fluorescence spectra were measured with a Hitachi F-4500 luminescence spectrometer.

1.2 Synthesis of the complex

An 6.0 mL ethanol solution was added to a solution of $AgNO_3$ (0.017 0 g, 0.1 mmol), H_2tsgluO (0.030 1 g, 0.1 mmol), and bpp (0.019 8 g, 0.1 mmol) in water (6.0 mL), and the mixture was stirred. The pH value of the above solution was then carefully adjusted to about 5.0 by slow addition of 2% aqueous triethylamine solution and the mixture was then filtered and the filtrate was allowed to stand at room temperature in the dark for thirty days. Concentration by slow evaporation led to the precipitation of colorless elongated crystals of **1** in low yield. Anal. Calcd. for $[Ag(HtsgluO)(bpp)]_n$ $C_{25}H_{28}AgN_3O_6S$(%): C, 49.51; H, 4.65; N, 6.93. Found (%): C, 49.44; H, 4.64; N, 6.94. FTIR (KBr pellet): 3 347(b), 2 931(m), 1 704(m), 1 605(s), 1 498(w), 1 419(m), 1 397(m), 1 324(m), 1 306(m), 1 223(w), 1 159(s), 1 094(m), 1 019(w), 902(w), 813(m), 707(w), 666(m), 567(m).

1.3 X-ray crystallographic study

Single crystals of dimensions 0.48 mm×0.17 mm×0.12 mm for **1** was used for structural determinations on a Bruker SMART APEX CCD area detector diffractometer using graphite-monochromatized Mo $K\alpha$ radiation (λ=0.071 073 nm) at room temperature according to the ω-φ scan technique. The structure was solved by direct methods using the SHELXS-97 program. All non-hydrogen atoms were refined

Table 1　Crystallographic data and refinement parameters for complex 1

Empirical formula	$C_{25}H_{28}AgN_3O_6S$	μ / mm^{-1}	0.905
Formula weight	606.43	F(000)	1240
Wavelength/nm	0.071073	Crystal size / mm	0.48×0.17×0.12
Crystal system	Monoclinic	θ range / (°)	0.99~25.50
Space group	$P2_1$	Reflections collected	13 707
a / nm	1.220 79(18)	Independent reflections (R_{int})	9 170 (0.027 2)
b / nm	1.034 49(16)	Max. and min. transmission	0.899 2, 0.670 6
c / nm	2.059 5(3)	Data / restraints / parameters	9 170 / 17 / 635
β / (°)	96.006(2)	Goodness-of-fit on F^2	1.035
V / nm^3	2.586 7(7)	R / wR (I>2σ(I))	0.060 2 / 0.133 4
Z	4	R / wR (all data)	0.072 3 / 0.141 3
D_c / (g·cm^{-3})	1.557	Largest diff. peak and hole / (e·nm^{-3})	666, −572

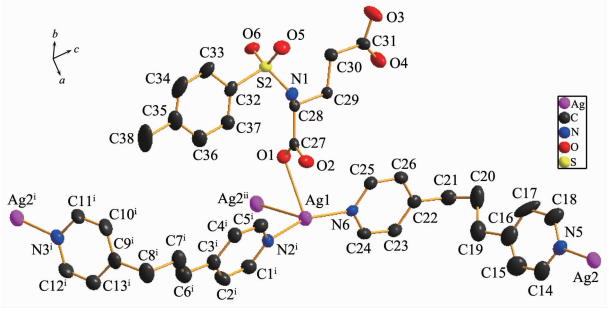
anisotropically by full-matrix least-squares methods on F^2 using the SHELXL-97 program[45]. The hydrogen atoms were added in geometrical positions and were not refined. A semi-empirical absorption correction was applied to the intensity data using SADABS. Complex **1** contains a disordered HtsgluO$^-$ ligand, which could be located at several sites between the layers. A summary of the crystallographic data and refinement parameters is given in Table 1.

CCDC: 795083.

2　Results and discussion

2.1　Crystal structure of the complex

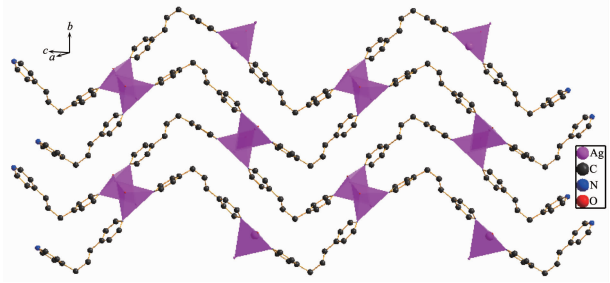
Reaction of H_2tsgluO and bpp with silver salts at room temperatuer, one homochiral complex [Ag (HtsgluO)(bpp)]$_n$ (**1**) suitable for single crystal X-ray diffraction analysis was obtained. The results revealed that complex **1** crystallizes in a chiral space group $P2_1$ and exhibits an infinite two-dimensional (2D) layered structure. The asymmetric unit contains one independent silver(I) ion, one HtsgluO$^-$ anion, and one bpp ligand. As illustrated in Fig.1, the silver (I) ion is located in a distorted tetrahedral environment formed by one oxygen atom from one HtsgluO$^-$, two nitrogen atoms from two different bpp ligands, and one symmetry-related silver(I) ions. The silver(I)-silver(I) separation (0.305 58 nm) observed in the unit is close to that of metallic silver (0.288 nm) and significantly less than twice the van der Waals radii for silver (0.344 nm), indicating the presence of an intramolecular argentophilic Ag⋯Ag interactions[46-49]. The Ag-O and Ag-N distances are in the range of 0.2510(6)~0.2611(5) nm and 0.216 5(7)~0.220 8(8) nm, and the angles of N-Ag-N, N-Ag-O, N-Ag-Ag, and O-Ag-Ag are about 160.7 (2)°~164.9 (2)°, 92.2 (3)°~104.7(2)°, 92.79(18)°~97.15(18)°, and 60.56(10)° ~111.17(17)°, respectively (selected bond lengths and angles are listed in Table 2). The C40, C41, C42, C43, O9, O10 atoms of HtsgluO$^-$ coordinated to Ag(2) are found to be disordered over two sites with partical occupancy at 60% and 40%.



Hydrogen atoms were omitted for clarity; Symmetry codes: ^{i} $x-1$, y, $z-1$; ^{ii} $-x+1$, $y+1/2$, $-z+1$

Fig.1　Coordination environment of silver(I) ions and HtsgluO$^-$ ligand in **1**

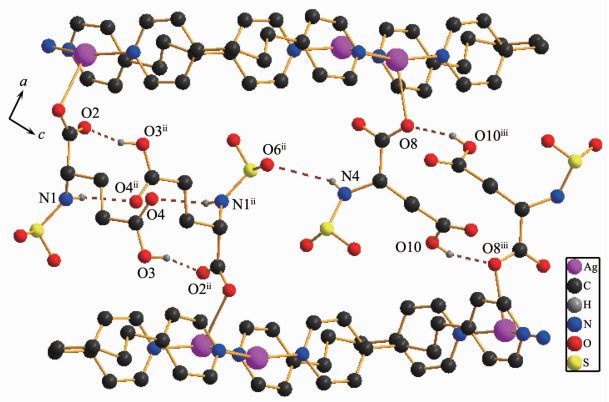
The bpp ligand shows a bis-monodentate mode with TG conformation providing a N-to-N separation with the distance of 0.873 7 or 0.864 0 nm and link the adjacent silver(I) ions to form a 1D zigzag chain. Then these 1D chains are further crosslinked by argentophilic Ag⋯Ag interactions generating a 2D layered network, as depicted in Fig.2. It is noteworthy



H_2tsgluO ligands and hydrogen atoms were omitted for clarity

Fig.2 2D layered network via argentophilic Ag···Ag interactions crosslinking 1D zigzag chains

that H_2tsgluO is monodeprotonated and coordinate to the silver(I) ions with the α-carboxyl group in a monodentate fashion, while the γ-carboxyl group remains protonated and does not take coordination with silver(I) ions. Therefore, the HtsgluO$^-$ ligands decorate such a 2D network as pendants at two sides in an outward fashion. However, to the best of our knowledge, the coordination mode adopted by the HtsgluO$^-$ anion has not been reported hitherto. Additionally, these unique 2D layers are further extended through extensive hydrogen bonding interactions between the uncoordinated γ-carboxyl groups or NH groups and the coordinated α-carboxyl groups and sulphonyl moieties to afford a final 3D supramolecular architecture, which consolidate the structure (Fig.3). Undoubtedly, the monodentate HtsgluO$^-$ anion are important in sustaining this structure. Details for all supramolecular hydrogen-bonding interactions in **1** are given in Table 3.



Interlayer hydrogen-bonding interactions are represented as dashed lines; the methylphenyl moieties in H_2tsgluO ligands were omitted for clarity; Symmetry codes: ii −x+1, y+1/2, −z+1; iii −x+1, y−1/2, −z+1

Fig.3 3D supramolecular array in complex **1**

To the best of our knowledge, $[Ag(HtsgluO)(bpp)]_n$ represents the first example of silver(I) homochiral coordination polymer constructed by H_2tsgluO and bridging pyridyl-containing ancillary ligand. Noticeably, although there are only one oxygen atom of the two carboxyl groups taking coordination with silver(I) ions, the rest uncoordinated carboxyl oxygen atoms

Table 2 Selected bond lengths (nm) and angles (°) for complex 1

Ag(1)-N(2)^{i}	0.218 2(7)	Ag(1)-N(6)	0.218 6(6)	Ag(1)-O(1)	0.261 1(5)
Ag(1)-Ag(2)^{ii}	0.305 58(10)	Ag(2)-N(3)	0.216 5(7)	Ag(2)-N(5)	0.220 8(8)
Ag(2)-O(8)	0.251 0(6)	Ag(2)-Ag(1)^{iii}	0.305 58(10)		
N(2)^{i}-Ag(1)-N(6)	160.7(2)	N(2)^{i}-Ag(1)-O(1)	104.7(2)	N(6)-Ag(1)-O(1)	93.85(18)
N(2)^{i}-Ag(1)-Ag(2)^{ii}	97.15(18)	N(6)-Ag(1)-Ag(2)^{ii}	96.60(16)	O(1)-Ag(1)-Ag(2)^{ii}	60.56(10)
N(3)-Ag(2)-N(5)	164.9(2)	N(3)-Ag(2)-O(8)	92.2(3)	N(5)-Ag(2)-O(8)	95.8(3)
N(3)-Ag(2)-Ag(1)^{iii}	92.79(18)	N(5)-Ag(2)-Ag(1)^{iii}	96.29(18)	O(8)-Ag(2)-Ag(1)^{iii}	111.17(17)

Symmetry codes: i x−1, y, z−1; ii −x+1, y+1/2, −z+1; iii −x+1, y−1/2, −z+1.

Table 3 Hydrogen-Bonding interactions in the complex

D–H···A	d(D-H) / nm	d(H···A) / nm	d(D···A) / nm	∠(DHA) / (°)
O(10)–H(10A)···O(8)^{v}	0.082	0.186	0.264 3(15)	159.7
O(3)–H(3)···O(2)^{vi}	0.082	0.182	0.253 2(7)	144.6
N(4)–H(4A)···O(6)^{vii}	0.086	0.251	0.316 9(7)	133.9
N(1)–H(1A)···O(4)^{vii}	0.086	0.212	0.288 8(7)	149.1

Symmetry codes: v −x+1, y−1/2, −z+2; vi −x, y+1/2, −z+1; vii −x, y−1/2, −z+1.

and nitrogen atoms of the $HtsgluO^-$ anion involve in the formation of hydrogen-bonding interactions, thus forming a stable chiral molecular configuration and transferring the chirality into the coordination polymer. The result has demonstrated that the chiral flexible H_2tsgluO ligands with the combined effect of the mixed flexible bridging ligands bpp are capable of adjusting themselves to adopt proper coordination modes to satisfy the needs for the formation of homochiral compounds. It proves that the usage of chiral ligands with flexible nature as the building unit are the more straightforward and effective approach to construct homochiral coordination polymers.

2.2 Thermal analysis

To characterize the complex more fully in terms of thermal stability, the thermogravimetric analysis (TGA) was performed from room temperature to 850 ℃ in an N_2 atmosphere (Fig.4). The TG curve shows two stages decomposition in the temperature range 113~728 ℃ and produces a 85.71% weight change, which corresponds to the release of H_2tsgluO and bpp ligands from the structure (calculated weight loss 82.21%).

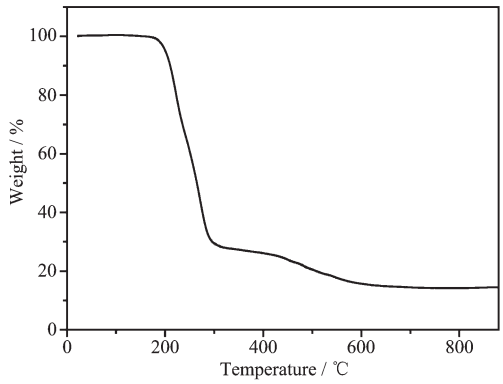


Fig.4 TGA curve for **1** in an N_2 atmosphere

2.3 Luminescent property

The silver (I) coordination compounds with aromatic ligands have received much attention for the development of hybrid photoluminescent materials[10,50-54]. Thus, photoluminescent properties of complex **1** as well as free ligands in ethanol solution were investigated at room temperature. Excitation of solid samples at λ=277 nm produces luminecescence peaks with maxima at 303 and 326 nm for complex **1** respectively. The free ligand bpp displays photoluminescence with emission maxima at 307 and 328 nm (λ_{ex}=280 nm) respectively. It can be presumed that the peaks originate from the π^*-n or π-π^* transitions. The photoluminescence spectra of the free ligand bpp and complex **1** are shown in Fig.5. The emission bands of complex **1** are similar to that of bpp and should be assigned to the intraligand transition of the coordinated *N*-donor ligand.

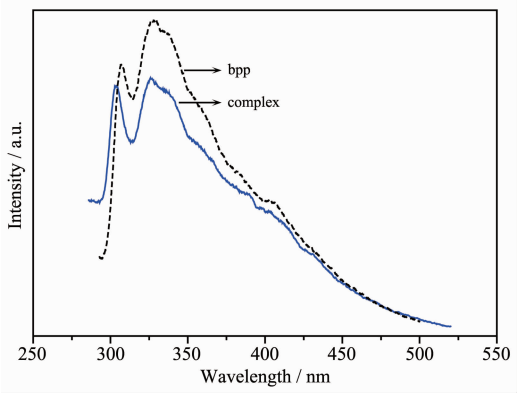


Fig.5 Photoluminescences of complex **1** and free bpp ligand

3 Conclusions

In summary, a novel homochiral coordination polymer $[Ag(HtsgluO)(bpp)]_n$ have been successfully synthesized at room temperature, by utilizing the flexible chiral H_2tsgluO ligand and the ancillary dipyridyl-containing bridging ligand bpp. The complex with a ligand unsupported Ag … Ag and hydrogen bonding interactions form a 3D supramolecular array. TGA measurement revealed that the polymer can be stable up to 113 ℃. It exhibits luminescence with emission maxima at 303 and 326 nm when excited at 277 nm. Noticeably, the monodentate coordination mode adopted by the $HtsgluO^-$ anion has not been reported in documents so far. Our research demonstrates that the chiral H_2tsgluO ligand could be a potential building unit with the combination of coligands to forming homochiral architectures with special properties.

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