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Synthesis, crystal structures, and photoluminescence of two silver(I) coordination polymers based on 2-sulfoterephthalic acid and N-donor ligands

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Abstract The reactions of 2-sulfoterephthalic acid (H_3stp) with silver nitrate in the presence of 2-aminopyrimidine (apym) or 2-amino-4,6-dimethylpyrimidine (dapym) generated two 2-D coordination polymers $\{[Ag_3(stp)(apym)_3]\cdot 2H_2O\}_n$ (**1**) and $\{[Ag_2(Hstp)(dapym)_2(H_2O)]\cdot H_2O\}_n$ (**2**). The complexes have been characterized by single-crystal X-ray diffraction, physico-chemical, and spectroscopic methods. Both complexes have a 2-D layer structure with infinite 1-D chains linked by stp^{3-} ligands and hydrogen bonds. The luminescent properties of the complexes were investigated.

Introduction

Reasons for the current interest in the crystal engineering of silver coordination polymers include their interesting mechanisms of molecular self-assembly [1–3], abundant weak interactions [4–6], potential applications in many areas such as electrical conductivity [7], magnetism [8], and catalysis [9] and so forth. Silver(I) atoms can accommodate a wide variety of stereochemistries, with coordination numbers of two to six and versatile coordination geometries, including linear, T-shaped, square-planar, tetrahedral, square-pyramidal, trigonal-bipyramidal, and octahedral [10–13]. Furthermore, Ag(I) with a d^{10} closed-shell electronic configuration tends to form argentophilic interactions, which have an important influence on the formation of polymetallic clusters [14–16]. In particular, Ag(I) complexes with carboxylate ligands have been widely investigated for their intriguing structural topologies and potential applications [17–20]. We selected 2-sulfoterephthalic acid as an organic ligand, based on the following twofold considerations. First, the two carboxylate groups and one sulfonate group of the ligand could act as bridging groups to form coordination polymers [21–24]; second, the protons of the ligand, if not all dissociated, could form H-bonds with adjacent ligands or solvent molecules [10, 25]. In addition, the introduction of N-donor organic ligands is an important strategy for the construction of novel coordination polymers [26–28].

Taking into account these considerations, and following on from our previous work [29], we have studied the self-assembly of Ag(I) with H_3stp and auxiliary N-donor ligands. In this paper, we report the syntheses, crystal structures, and properties of two new silver(I) coordination polymers, namely $\{[Ag_3(stp)(apym)_3]\cdot 2\ H_2O\}_n$ (**1**) and $\{[Ag_2(Hstp)(dapym)_2(H_2O)]\cdot H_2O\}_n$ (**2**).

Experimental

Materials and instrumentation

All chemicals and solvents were obtained from commercial sources as reagent grade and used without further purification in the syntheses. Elemental analyses for C, H, and N were obtained with a CHN-O-Rapid Analyzer and an Elemental Vario Microanalyzer. The infrared spectra were taken on a Bruker Equinox 55 FTIR spectrometer as KBr pellets in the 400–4,000/cm^{-1} region. The fluorescence spectra were measured using a Shimadzu RF-7000 spectrometer on powdered samples in the solid state at room temperature.

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Synthesis of $\{[Ag_3(stp)(apym)_3]\cdot 2H_2O\}_n$ ***(1)***

A solution of $AgNO_3$ (0.0338 g, 0.20 mmol) in water (10 ml) was added to a stirred solution of 2-sulfotereph-thalic acid (0.0536 g, 0.20 mmol) and 2-aminopyrimidine (0.019 g, 0.20 mmol) in DMF(5 ml). The mixture was stirred for 10 min and then the precipitate was filtered off; the filtrate was allowed to evaporate in the dark. After 2 weeks, colorless block-shaped crystals were obtained. Yield 0.07 g (40 %). Anal. Calcd for $C_{20}H_{22}Ag_3N_9O_9S$: C 27.1, H 2.5, N 14.2 %; Found: C 27.0, H 2.4, N 14.1 %; FTIR (KBr pellet, cm^{-1}) selected bands: v = 3,856 w, 3,740 w, 3,403 s, 3,314 s, 3,178 m, 1,577 s, 1,479 s, 1,360 s, 1,203 s, 1,072 m, 1,022 m, 799 m, 676 w, 621 m, 574 w, 508 m.

Synthesis of $\{[Ag_2(Hstp)(dapym)_2(H_2O)]\cdot H_2O\}_n$ ***(2)***

A solution of $AgNO_3$ (0.0338 g, 0.20 mmol) in water (10 ml) was added to a stirred solution of 2-sulfotereph-thalic acid (0.0536 g, 0.20 mmol) and 2-amino-4,6-dim-ethylpyrimidine (0.0246 g, 0.20 mmol) in a mixture of DMF(5 ml) and CH_3CN(5 ml). The mixture was stirred for 10 min and then the precipitate was filtered off; the filtrate was allowed to evaporate in the dark. After 2 weeks, col-orless sheet crystals were obtained. Yield 0.06 g (38 %). Anal. Calcd for $C_{20}H_{26}Ag_2N_6O_9S$: C 32.4, H 3.5, N 11.3 %; Found: C 32.3, H 3.5, N 11.3 %; FTIR (KBr pellet, cm^{-1}) selected bands: v = 3,856, 3,741 w, 3,508 m, 3,393 s, 3,213 m, 1,937 m, 1,647 s, 1,589 s, 1,475 m, 1,427 m, 1,372 s, 1,285 m, 1,223 s, 1,074 m, 1,023 m, 789 m, 728 w, 672 w, 624 m, 572 w, 496 m.

Table 1 Crystal data for complexes **1** and **2**

Complex	**1**	**2**
Empirical formula	$C_{20}H_{22}Ag_3N_9O_9S$	$C_{20}H_{26}Ag_2N_6O_9S$
M_r	888.14	742.27
Crystal system	Triclinic	Monoclinic
Space group	P_{-1}	$P\ 2_1/c$
a (Å)	8.4598(5)	9.8955(14)
b (Å)	8.7939(5)	22.883(3)
c (Å)	18.5836(11)	13.5489(13)
α (°)	80.2270(10)	90
β (°)	81.5540(10)	124.005(7)
γ (°)	74.6730(10)	90
V (Å^3)	1,306.41(13)	2,543.3(5)
Z	2	4
θ range (°)	1.12–25.10	2.02–25.50
D_{calc}(Mg m^{-3})	2.258	1.939
μ(mm^{-1})	2.377	1.684
F(000)	868	1,480
R_1 [I > 2σ]	0.0287	0.0308
wR_2 (all data)	0.0361	0.0325

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

X-ray crystallographic study

The X-ray single-crystal data for complexes **1** and **2** were recorded on a Brucker APEX II area detector diffractom-eter with graphite-monochromated MoKα radiation (k = 0.71073 Å). Semi-empirical absorption corrections were applied using the SADABS program. The structures were solved by direct methods and refined by full-matrix least squares on F^2 using SHELXL-97. All non-hydrogen atoms were refined anisotropically. The carboxyl and water H atoms were located from difference Fourier maps; other hydrogen atoms were placed in geometrically calculated positions. Experimental details for X-ray data collection of **1** and **2** are presented in Table 1, and selected bond lengths are listed in Table 2.

Results and discussion

Structure of complex **1**

Single-crystal X-ray diffraction analysis reveals that $\{[Ag_3(stp)(apym)_3]\cdot 2H_2O\}_n$ (**1**) presents a 2-D sheet based on a 1-D double chain, which crystallizes in the space group P_{-1}. There are three Ag(I) atoms, one 2-stp ligand, three apym ligands, and two lattice water molecules in the asymmetric unit of **1**. As depicted in Fig. 1a, three crys-tallographically different Ag(I) atoms are located in an approximate T-shaped coordination geometry and coordi-nated by one oxygen from the same 2-stp ligand and two nitrogen atoms from two different apym ligands. The Ag–O bond distances vary from 2.283(3) to 2.509(3) Å, and the Ag–N bond lengths range between 2.190(3) and 2.330(4) Å. Both Ag–O and Ag–N bond lengths are well matched with those observed in similar complexes [10–14, 29].

The Ag(I) atoms are linked by bidentate apym ligands to form 1-D zigzag chains in which the apym ligands are oppositely arranged, and the stp ligands extend the 1-D single chains into a 1-D double chain, containing classical hydrogen bonds (Fig. 1c). Subsequently, with Ag2 as a node, two pairs of reverse direction 1-D double chains link adjacent sulfo group oxygen atoms (O3) from stp ligands into a 2-D laminar framework (Fig. 1b). In addition, the amino groups of apym ligands form hydrogen bonds with the coordinated O5 of the carboxyl groups and water molecules, while the water molecule form hydrogen bonds with the coordinated O2 and O5 atoms of the carboxyl groups (as depicted in Table 3).

Table 2 Selected bond lengths (Å) angles (°) for complexes **1** and **2**

$\{[Ag_3(stp)(apym)_3]\cdot 2H_2O\}_n$ (**1**)					
Bond lengths (Å)					
Ag(1)-O(1)	2.499(3)	Ag(1)-N(1)	2.206(3)	Ag(1)-N(9)#1	2.190(3)
Ag(2)-O(3)	2.509(3)	Ag(2)-N(7)	2.233(3)	Ag(3)-O(6)	2.283(3)
Ag(3)-N(3)#2	2.226(3)	Ag(3)-N(6)#3	2.330(4)		
Bond angles (°)					
N(9)#1-Ag(1)-N(1)	157.91(12)	N(9)#1-Ag(1)-O(1)	98.97(12)	N(1)-Ag(1)-O(1)	102.85(12)
N(7)-Ag(2)-N(4)	153.11(13)	N(7)-Ag(2)-O(3)	97.28(11)	N(4)-Ag(2)-O(3)	108.11(11)
O(6)-Ag(3)-N(6)#3	98.35(12)	N(3)#2-Ag(3)-N(6)#3	124.68(12)	N(3)#2-Ag(3)-O(6)	132.10(12)
Symmetry codes: #1($-x+2,-y+1,-z$); #2($-x+2,-y,-z+1$); #3($x, y-1, z$)					
$\{[Ag_2(Hstp)(dapym)_2(H_2O)]\cdot H_2O\}_n$ (**2**)					
Bond lengths (Å)					
Ag(1)-N(4)	2.200(3)	Ag(1)-N(1)	2.202(3)	Ag(1)-O(1)	2.494(2)
Ag(2)-O(8)	2.444(3)	Ag(2)-O(4)	2.715(6)	Ag(2)-N(2)#1	2.250(3)
Ag(2)-N(5)#2	2.285(3)				
Bond angles (°)					
N(4)-Ag(1)-N(1)	157.36(9)	N(1)-Ag(1)-O(1)	99.04(9)	N(4)-Ag(1)-O(1)	96.96(9)
O(8)-Ag(2)-O(4)	99.34(19)	N(2)#1-Ag(2)-O(8)	100.50(10)	N(5)#2-Ag(2)-O(8)	94.24(10)
N(2)#1-Ag(2)-O(4)	112.97(14)	N(5)#2-Ag(2)-O(4)	93.80(14)	N(2)#1-Ag(2)-N(5)#2	150.79(9)
Symmetry codes: #1($x,-y+3/2,z+1/2$); #2($-x,-y+1,-z+1$)					

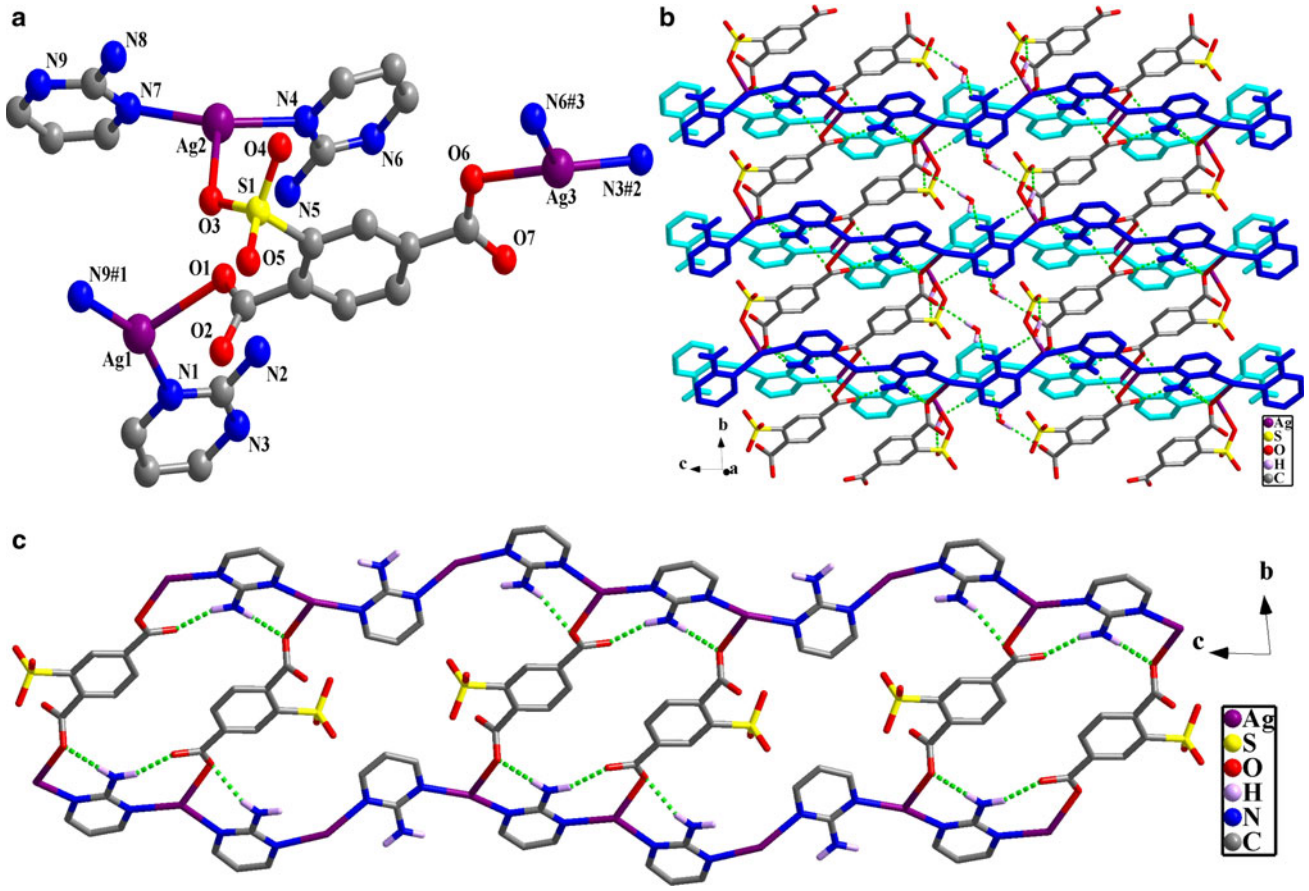

Fig. 1 **a** Asymmetric unit of $\{[Ag_3(stp)(apym)_3]\cdot 2H_2O\}_n$ (**1**) and coordination environments around the Ag(I) atoms. Water molecules and corresponding H atoms are omitted for clarity. **b** The 2-D laminar structure in complex **1** based on the Ag(I) atoms, stp and apym ligands along $a \times b \times c$ plane. **c** View of the 1-D double chain in complex **1** along the a axis

Table 3 Hydrogen bonds for complexes 1 and 2 (Å and °)

D-H···A	d(D-H)	d(H···A)	<(DHA)	d(D···A)
$\{[Ag_3(stp)(apym)_3]\cdot 2H_2O\}_n$ (**1**)				
N(8)-H(8B)···O(8)	0.90	1.96	173.0	2.852(5)
N(8)-H(8A)···O(5)#5	0.90	2.64	132.3	3.312(6)
N(8)-H(8A)···O(9)#6	0.90	2.38	125.8	2.993(7)
N(5)-H(5B)···O(1)	0.90	2.16	133.6	2.853(5)
N(5)-H(5A)···O(6)#4	0.90	2.13	148.7	2.934(5)
N(2)-H(2B)···O(7)	0.90	2.03	160.1	2.890(5)
N(2)-H(2A)···O(1)	0.90	2.08	177.0	2.983(5)
O(9)-H(9C)···O(2)#7	0.85	2.33	159.0	3.142(7)
O(8)-H(8E)···O(4)	0.85	2.12	132.6	2.769(5)
O(8)-H(8C)···O(2)#8	0.85	2.02	130.0	2.651(5)
Symmetry codes: #4 $x, y + 1, z$; #5 $-x + 2,-y,-z$; #6 $-x + 1,-y + 1,-z$; #7 $x-1,y + 1, z$; #8 $x-1,y,z$				
$\{[Ag_2(Hstp)(dapym)_2(H_2O)]\cdot H_2O\}_n$ (**2**)				
N(6)-H(6B)···O(1)	0.86	2.39	136.3	3.072(4)
N(6)-H(6B)···O(3)	0.86	2.33	138.5	3.027(7)
N(6)-H(6B)···O(3′)	0.86	2.09	139.6	2.802(7)
N(6)-H(6A)···O(4′)#2	0.86	1.99	167.0	2.838(7)
N(6)-H(6A)···O(4)#2	0.86	1.91	168.7	2.757(6)
N(3)-H(3B)···O(1)	0.86	2.54	137.7	3.224(4)
N(3)-H(3A)···O(8)#3	0.86	2.07	163.0	2.905(4)
O(9)-H(9C)···N(4)#4	0.85	2.61	148.5	3.366(5)
O(9)-H(9B)···O(3)#4	0.85	2.18	118.5	2.696(7)
O(8)-H(8C)···O(9)	0.85	1.98	151.4	2.761(5)
O(8)-H(8B)···O(2)	0.85	1.88	154.5	2.671(4)
O(7)-H(7)···O(2)#6	0.82	2.65	128.2	3.221(4)
O(7)-H(7)···O(1)#6	0.82	1.76	171.7	2.577(3)
Symmetry codes: #2$-x,-y + 1,-z + 1$; #3 $x,-y + 3/2, z-1/2$; #4 $-x + 1, y + 1/2,-z + 1/2$; #6$x + 1,-y + 3/2, z + 1/2$				

Crystal structure of complex **2**

The complex **2** crystallizes in the monoclinic $P2_1/c$ space group. The asymmetric unit of **2** contains two Ag(I) atoms, one Hstp ligand, two dapym ligands, and one aqua ligand, as well as one lattice water molecule. As illustrated in Fig. 2a, the Ag1 adopts a *T*-shaped geometry completed by one oxygen from the Hstp ligand and two nitrogen atoms from different dapym ligands. The maximum angle around Ag1 is 157.36(9) Å. Atom Ag2 is located in a distorted tetrahedral geometry with one aqua ligand, one oxygen atom from an Hstp ligand, and two nitrogen atoms from two dapym ligands as donors. Each dapym ligand uses the μ_2-η^1:η^1 bidentate coordination mode to bridge two adjacent Ag(I) atoms. As a consequence, the Ag(I) atoms are extended by the dapym ligand spacers to afford a 1-D chain almost along the [001] direction (Fig. 2c), within which the distance between successive Ag(I) atoms is 6.142(9) Å. The Hstp ligand acts as a linker to equip a 2-D layer (Fig. 2b) by connecting two neighboring parallel coordination chains through Ag–O coordination, as well as intermolecular hydrogen bond interactions with the coordinated carboxylate and sulfo group oxygen atoms of Hstp ligands, plus further H-bonding involving both coordinated and guest water, as depicted in Table 3.

Thermogravimetric and photoluminescence analyses

Thermogravimetric (TG) analysis was performed under an N_2 atmosphere on crystalline samples of complexes **1** and **2** (Fig. 3a). The TG curve of **1** shows the first weight loss of 4.12 % in the temperature range 40–80 °C, associated with the exclusion of two lattice water molecules (calcd 4.85 %); then, from 200 to 700 °C, the stp and apym ligands are released in a featureless process, to give a residue of Ag_2SO_4. Complex **2** starts to lose one lattice water molecule in the temperature range 40–80 °C (obsd 1.74 %; calcd 2.02 %), and then, the curve shows further decomposition in the temperature range 200–250 °C with a

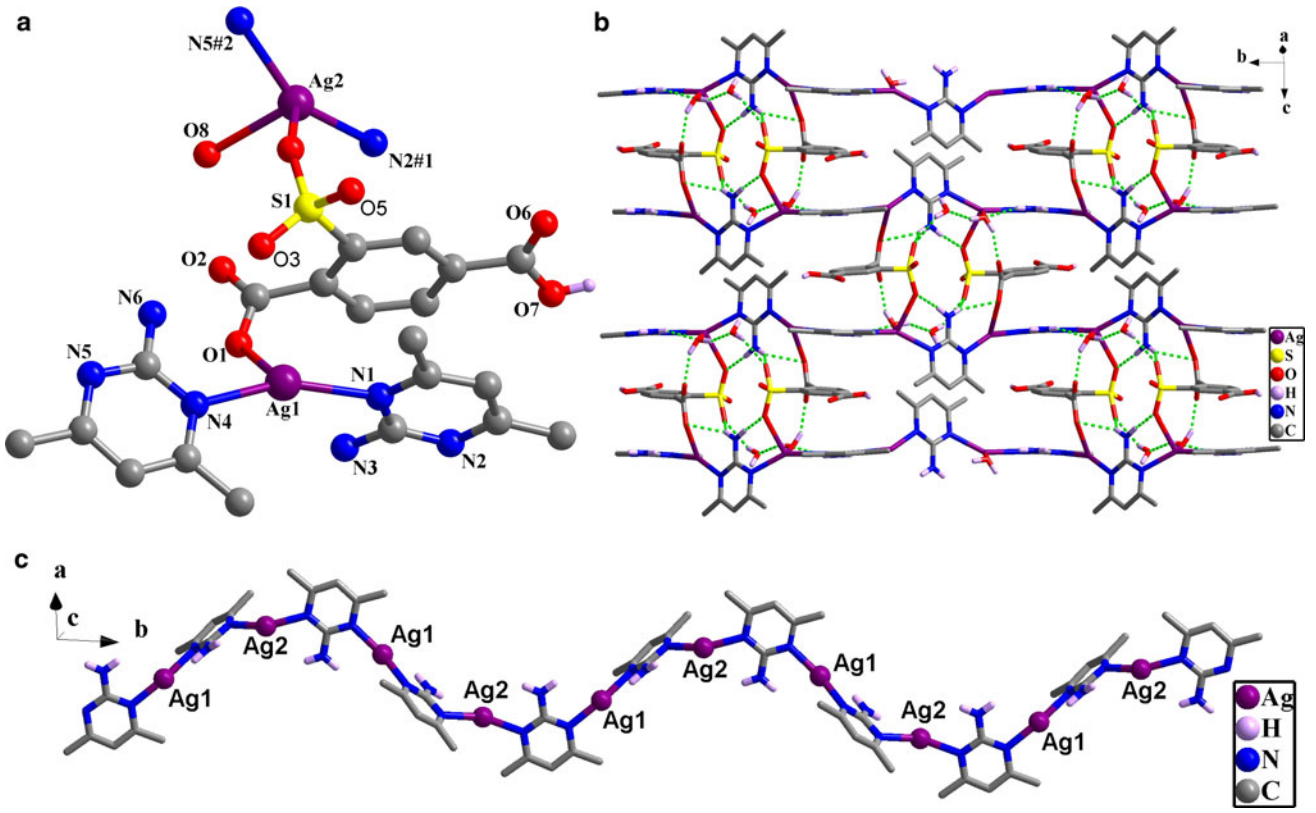

Fig. 2 **a** Asymmetric unit of $\{[Ag_2(Hstp)(dapym)_2(H_2O)]\cdot H_2O\}_n$ (**2**) and coordination environments around the Ag(I) atoms. Water molecules and corresponding H atoms are omitted for clarity. **b** 2-D layer structure in complex **2** consisting of hydrogen bonds. **c** 1-D metal-organic polymer based on the Ag(I) atoms and dapym ligands

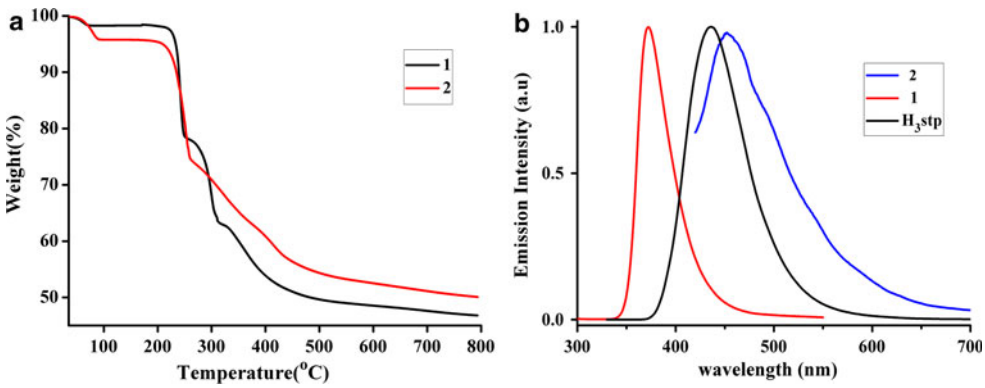

Fig. 3 **a** TGA curves for complexes **1** and **2**. **b** The solid-state emission photoluminescent spectra of **1** ($\lambda_{ex} = 390$ nm), **2** ($\lambda_{ex} = 290$ nm) and free H_3stp ($\lambda_{ex} = 321$ nm) at room temperature

20.00 % weight change, corresponding to loss of the dapym ligand (calcd 20.08 %). The third weight loss of 13.72 % (calcd 12.72 %) corresponds to the loss of the Hstp ligands and one aqua ligand without a clear inflection.

The luminescence properties of both complexes and free H_3stp were studied in the solid state. As shown in Fig. 3b, very similar emission bands were observed for the two complexes, while emission of the free H_3stp ligand with $\lambda_{em} = 437$ nm ($\lambda_{ex} = 321$ nm) was observed, which is probably attributable to $\pi^* \rightarrow n$ or $\pi^* \rightarrow \pi$ transitions. Compared to the spectrum of the free ligand, the emission band of complex **1** is red-shifted by 15 nm. The shift can be attributed to ligand to metal charge transfer (LMCT), involving the filled p orbitals of coordinated N atoms and the vacant 5 s orbitalof Ag(I), mixed with metal-centered (d–s/d–p) transitions [30–32]. In contrast to complex **1**, the emission band of **2** is blue-shifted by more than 65 nm, which could be assigned to $\pi^* \rightarrow \pi$ electronic transitions of the ligand. The blue shift of the emission compared with that of the free ligand may be attributed to coordination of the ligand to Ag(I), enhancing its conformational rigidity and decreasing the non-radiative energy loss [12].

Generally speaking, Ag(I) complexes emit weak photoluminescence at low temperature, owing to the intense spin-orbital coupling of Ag(I) [12, 33, 34]. Consequently, complexes **1** and **2** are unusual examples of room temperature luminescent Ag-containing polymers.

Conclusions

From the reactions of $AgNO_3$, 2-sulfoisophthalic acid, and N-donor ligands, we synthesized and structurally characterized two Ag(I) coordination complexes, in which the auxiliary ligands are different. This investigation may supply some new information for the design and crystal engineering of such crystalline materials. In addition, these complexes display modest thermal stability and solid-state fluorescent emission. Further studies in this respect are under way in our laboratory.

Supplementary material

CCDC 912814, 912815 contain the supplementary crystallographic data for complexes **1** and **2**. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via http://www.ccdc.cam.ac.uk/deposit or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK [Telephone: +44-01223-762910; Fax:+44-01223-336033; E-mail: deposit@ccdc.cam.ac.uk].

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