



Dimeric and polymeric silver(I) saccharinato complexes of two bis(pyridine) ligands: Synthesis, structural, spectroscopic, fluorescent and thermal properties

Emel Guney^a, Veysel T. Yilmaz^{a,*}, Orhan Buyukgungor^b

^a Department of Chemistry, Faculty of Arts and Sciences, Uludag University, 16059 Bursa, Turkey

^b Department of Physics, Faculty of Arts and Sciences, Ondokuz Mayıs University, 55139 Samsun, Turkey

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ABSTRACT

The reaction of silver(I) with 1,2-bis[1-(pyridin-2-yl)ethylidene]hydrazine (bpeh) and *N,N*-bis(pyridin-2-ylmethyl)amine (bpma) in the presence of Na(sac) (sac = saccharinate) yielded $[\text{Ag}_2(\text{sac})_2(\text{bpeh})]$ (**1**) and $[\text{Ag}(\text{sac})(\text{bpma})]_n$ (**2**) with conformational chirality. Both complexes have been characterized by elemental analysis, IR, thermal analysis and X-ray single crystal diffraction. Complex **1** displays a binuclear composition, in which each silver(I) ion is bound to one monodentate sac ligand and one of the bidentate pyridylimino groups of the bpeh ligand in a distorted trigonal coordination geometry. Complex **2** is a one-dimensional helical polymer, in which silver(I) centers are bridged by tridentate bpma ligands, and each silver(I) ion is coordinated in a distorted tetrahedral geometry by one monodentate sac ligand, a bidentate pyridylamine group of one bpma ligand, and a py group of another bpma ligand. Weak intermolecular C–H···O hydrogen bonds and C–H··· π interactions lead to assembly of **1** and **2** into three-dimensional supramolecular frameworks. Spectral and thermal analysis data for **1** and **2** are in agreement with the crystal structures. In addition, both complexes in the solid state display intraligand π – π^* fluorescence.

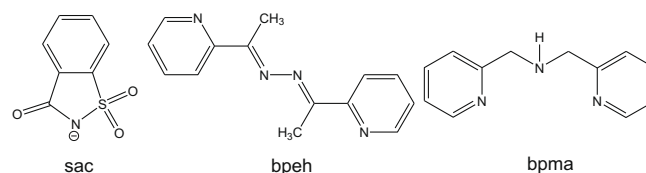
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1. Introduction

Saccharin (sacH) is a well-known and the most widely used sweetening agent. The imino hydrogen of sacH is readily deprotonated in solutions to give the corresponding monoanion sac. Following the discovery of the coordination ability of the sac anion towards biologically important metal ions, different metal complexes of sac in the presence of various co-ligands have been extensively studied. Earlier work in this field has been summarized in a recent review [1]. The sac ligand has several potential donor atoms such as an imino nitrogen, a carbonyl and two sulfonyl oxygen atoms. It usually coordinates in a monodentate fashion through the negatively charged N atoms and also acts as a bi- or tridentate bridging ligand, forming complexes from mononuclear to polymeric chains [1].

As continuation of our interest in the synthesis and properties of metal complexes of the sac ligand, we synthesized two new silver(I) complexes of sac with 1,2-bis[1-(pyridin-2-yl)ethylidene]hydrazine (bpeh) and *N,N*-bis(pyridin-2-ylmethyl)amine (bpma, alternatively named bis(2-picolyl)amine) as co-ligands, namely $[\text{Ag}_2(\text{sac})_2(\text{bpeh})]$ (**1**) and $[\text{Ag}(\text{sac})(\text{bpma})]_n$ (**2**). Both complexes have been characterized by elemental analyses and IR

studies. The crystal structures of the complexes were determined by X-ray diffraction. Complex **1** is obtained in a dinuclear form, while complex **2** adopts a one-dimensional helical zig-zag polymeric chain. Some solid state properties of the complexes such as thermal decomposition and photoluminescence were also studied.



2. Experimental

2.1. Materials and measurements

1,2-Bis[1-(pyridin-2-yl)ethylidene]hydrazine (bpeh) were prepared according to the reported method by the reaction of hydrazine hydrate with 2-acetylpyridine [2]. Other chemicals were purchased and used as supplied. Elemental analyses for C, H, and N were performed using a Costech elemental analyser. IR spectra were recorded with a Thermo Nicolet 6700 FTIR spectrophotometer

* Corresponding author.

E-mail address: vtyilmaz@uludag.edu.tr (V.T. Yilmaz).

Table 1
Crystallographic data and structure refinement for **1** and **2**.

	1	2
Formula	C ₂₈ H ₂₂ Ag ₂ N ₆ O ₆ S ₂	C ₁₉ H ₁₇ AgN ₄ O ₃ S
Molecular weight	818.38	489.30
T (K)	296	296
Wavelength (Å)	0.71073	0.71073
Crystal system	orthorhombic	orthorhombic
Space group	Ab a 2	P2 ₁ 2 ₁ 2 ₁
<i>Unit cell dimensions</i>		
<i>a</i> (Å)	14.4445(11)	7.7968(3)
<i>b</i> (Å)	25.6574(14)	9.8671(5)
<i>c</i> (Å)	7.7734(5)	24.8118(12)
Volume (Å ³)	2880.9(3)	1908.82(15)
<i>Z</i>	4	4
Calculated density (g/cm ³)	1.887	1.708
μ (mm ⁻¹)	1.560	1.194
<i>F</i> (0 0 0)	1624	784
Crystal size (mm ³)	0.36 × 0.23 × 0.03	0.58 × 0.35 × 0.11
θ Range (°)	1.59–26.49	1.64–26.50
Index ranges (<i>h</i> , <i>k</i> , <i>l</i>)	–9/16; –31/32; –9/5	–9/7; –11/12; –30/31
Reflections collected	5537	7132
Independent reflections	2375 [<i>R</i> _{int} = 0.0271]	3918 [<i>R</i> _{int} = 0.0404]
Reflections observed (>2 σ)	1981	3461
Absorption correction	numerical	numerical
Data/parameters	2375/200	3918/253
Flack parameter	0.39(4)	–0.03(3)
Goodness-of-fit on (GOF) <i>F</i> ²	0.977	1.056
Final <i>R</i> indices [<i>I</i> > 2 σ (<i>I</i>)]	<i>R</i> ₁ = 0.0321, <i>wR</i> ₂ = 0.0750	<i>R</i> ₁ = 0.0316, <i>wR</i> ₂ = 0.0747
Largest difference in peak and hole (e Å ⁻³)	1.100 and –0.423	0.472 and –0.659

with samples as KBr pellets in the 4000–400 cm⁻¹ range. Excitation and emission spectra were recorded at room temperature with a Varian Cary Eclipse spectrophotometer equipped with a Xe pulse lamp of 75 kW. Thermal analysis curves (TGA and DTA) were obtained using a Seiko Exstar 6200 thermal analyzer in a dynamic air atmosphere with a heating rate of 10 °C min⁻¹ and a sample size of ca. 10 mg.

2.2. Synthesis of the silver(I) complexes

The addition of Na(sac)·2H₂O (1 mmol, 0.24 g) dissolved in 5 ml water to a solution of AgNO₃ (0.17 g, 1 mmol) in 5 ml water resulted in a white suspension, which is dissolved by the addition of MeCN (5 ml). Then, a solution 1,2-bis[1-(pyridin-2-yl)ethylidene]hydrazine (bpeh) (0.5 mmol, 0.12 g) in 5 ml MeOH was added dropwise to the clear solution with stirring. Then, a pale yellow suspension was formed and clarified by addition of 10 ml MeCN and 10 ml isopropanol. The final solution was kept in darkness at room temperature. Pale yellow crystals of [Ag₂(sac)₂(bpeh)] (**1**) were obtained within a week. Yield 87.0%. M.p. 218–223 °C (with decomp.). *Anal.* Calc. for C₂₈H₂₂Ag₂N₆O₆S₂: C, 41.1; H, 2.7; N, 10.3. Found: C, 40.9; H, 2.9; N, 10.5%. IR (Solid KBr pellet): ν (cm⁻¹) 3052vw, 3003vw, 2952vw, 1652vs, 1626vs, 1584s, 1462m, 1430w, 1360w, 1340w, 1271vs, 1252vs, 1168vs, 1154vs, 1115sh, 1054m, 964m, 952m, 772m, 743m, 670m, 637w, 605m, 543m.

[Ag(sac)(bpma)]_n (**2**) was synthesized in a similar way, by replacing the ligand bpeh with *N,N*-bis(pyridin-2-ylmethyl)amine (bpma). Yield 90.0%. M.p. 189–192 °C. *Anal.* Calc. for C₁₉H₁₇AgN₄O₃S (%): C, 46.6; H, 3.5; N, 11.5. Found: C, 46.3; H, 3.6; N, 11.7%. IR (cm⁻¹): 3281m, 3060vw, 2923vw, 2848vw, 1659sh, 1626vs, 1592s, 1477w, 1435w, 1336w, 1259vs, 1152vs, 1120sh, 1052w, 1005vw, 953s, 850w, 761s, 677w, 636w, 604m, 543m.

2.3. X-ray crystallography

The intensity data of the complexes **1** and **2** were collected using a STOE IPDS 2 diffractometer with graphite-monochromated

Mo K α radiation (λ = 0.71073 Å). The structures were solved by direct methods and refined on *F*² with the SHELX-97 program [3]. All non-hydrogen atoms were found from the difference Fourier map and refined anisotropically. All hydrogen atoms were included using a riding model. The details of data collection, refinement and crystallographic data are summarized in Table 1.

3. Results and discussion

3.1. Synthesis and characterization

Complexes **1** and **2** were synthesized by the direct reaction of Na(sac) with AgNO₃ in the aqueous solution in the presence of the bpeh and bpma ligands. They were obtained in high yields (over 85%). Both complexes are air stable and soluble in MeCN and a mixture of water, PrⁱOH and MeCN (1:1.1).

Selected FTIR spectroscopic data for **1** and **2** are listed in Table 2. The azine group (C=N) of bpeh and the NH group of bpma have prominent IR bands centered at 1684 and 3281 cm⁻¹. The CN band of bpeh seems to couple with the ν (C=O) band of sac, leading to a most intense band centered at 1626 cm⁻¹. The sac moiety gives characteristic IR bands due to the absorption of the carbonyl and sulfonyl groups. The carbonyl O atom of the sac ligand does not

Table 2
Selected FTIR spectral data^a for **1** and **2**.

Assignment	1	2
ν (NH)		3281m
ν (CH)	3052vw, 3003vw, 2952vw	3060vw, 2923vw, 2848vw
ν (CO)	1652vs, 1626vs ^b	1659sh, 1626vs
ν (CC)	1584vs, 1462m	1592s, 1477w
ν _s (CNS)	1340w	1336w
ν _{as} (SO ₂)	1271vs	1259vs
ν _s (SO ₂)	1154vs	1152vs
ν _{as} (CNS)	964m	953s

^a Frequencies in cm⁻¹. b = broad; m = medium; w = weak; vw = very weak; vs = very strong; s = strong.

^b Coupled with the ν (C=N) vibration of bpeh.

participate in metal bonding and produce two sharp bands at around 1650 and 1625 cm^{-1} . The asymmetric (ν_{as}) and symmetric (ν_{s}) stretchings of the SO_2 group in the spectra of **1** and **2** are observed as two strong IR bands at ca. 1265 and 1150 cm^{-1} , respectively. Moreover, the ν_{s} and ν_{as} stretching modes of the CNS fragment of the sac ligands appear at 1340 and 955 cm^{-1} , respectively.

3.2. Crystal structures

A view of the molecular structure of complex **1** is shown in Fig. 1, together with the atom numbering scheme. Selected bond distances and angles are listed in Table 3. Complex **1** is a binuclear silver(I) complex consisting of two sac and one bpeh ligand. The bpeh ligand acts as a bis(bidentate) (tetradentate in total) ligand coordinating two silver(I) ions through both pyridimine groups. The distorted trigonal coordination sphere around each silver(I) ion is completed by a N-bonded sac ligand. The bpeh ligand is flexible, twisting about the central N–N bond. Therefore, it exhibits several stereoisomers such as *E–E* (*trans, trans*), *Z–E* (*cis, trans*), and *Z–Z* (*cis, cis*) [4]. In **1**, the dihedral angle between the py rings of bpeh is 54.65(13)° and the ligand adopts the *E–E* arrangement. On the other hand, it has been stated that binuclear composition of **1** is consistent with the *E–E* isomer of the ligand [5]. The bpeh ligand is known to generate helical silver(I) complexes with non-coordinating counter-ions, formulated as $\{[\text{Ag}_2(\text{bpeh})_2]\text{X}_2\}_n$ ($\text{X} = \text{ClO}_4^-$, BF_4^- and NO_3^-) [2]. In the present case, the ligand forms a binuclear complex of **1**. It has been explained that upon coordination as a bridge, the freedom of the ligand to rotate about the N–N bond is restrained and as a result, the bpeh ligand can be locked in a twisted chiral conformation [6]. Thus, the binuclear complex **1** is also chiral. Moreover, the Flack parameter of 0.39(4) indicates that the structure is racemically twinned, containing a partial mix of enantiomers. The Ag–N(bpeh) bond distances are similar to those of the complexes of the same or similar ligands [2,7], while the Ag–N(sac) bond distance of 2.134(4) Å is within the normal range observed in the reported N-bonded sac complexes of silver(I) [8–15]. It is interesting to note that no obvious π – π stacking interactions between the py rings of bpeh in the adjacent molecules are present. Thus, the dimeric

Table 3
Selected bond lengths [Å] and angles [°] for **1** and **2**.

	1	2
Ag1–N1	2.134(4)	2.303(3)
Ag1–N2	2.291(4)	2.396(3)
Ag1–N3	2.324(4)	2.360(3)
Ag1–N4		2.279(3)
N1–Ag1–N2	136.69(15)	109.36(13)
N1–Ag1–N3	150.02(14)	120.31(12)
N1–Ag1–N4		98.06(12)
N2–Ag1–N3	70.52(14)	73.20(12)
N2–Ag1–N4		130.37(11)
N3–Ag1–N4		126.14(12)

Table 4
Non-conventional hydrogen bonds in **1** and **2**.

D–H...A	D–H (Å)	H...A (Å)	D...A (Å)	D–H...A (°)
1 ^a				
C2–H2...O2 ⁱ	0.93	2.44	3.206(7)	140
C14–H14B...O3 ⁱⁱ	0.96	2.41	3.160(8)	134
C14–H14C...O1 ⁱⁱⁱ	0.96	2.47	3.330(5)	149
2 ^{a,b}				
C5–H5...O3 ^{iv}	0.93	2.53	3.436(5)	163
C8–H8...O1 ^v	0.93	2.48	3.151(6)	129
C13–H13A...O1 ^{vi}	0.97	2.54	3.401(6)	148
C2–H2...Cg1 ^{vii}	0.93	3.35	3.064	134
C9–H9...Cg2 ^{viii}	0.93	3.13	3.081	162

^a Symmetry codes: (i) $-x+2, -y+3/2, z-1/2$; (ii) $-x+2, -y+1, z+1$; (iii) $x+1/2, -y+1, z+1/2$; (iv) $x-1, y, z$; (v) $x+1, y, z$; (vi) $x+1/2, -y+1/2, -z+1$; (vii) $-x+1, y+1/2, -z+1/2$; (viii) $x-1/2, -y+5/2, -z$.

^b Cg1 and Cg2 are the centroids of the pyridine rings (N4, C14–C18) and (N2, C8–C12), respectively.

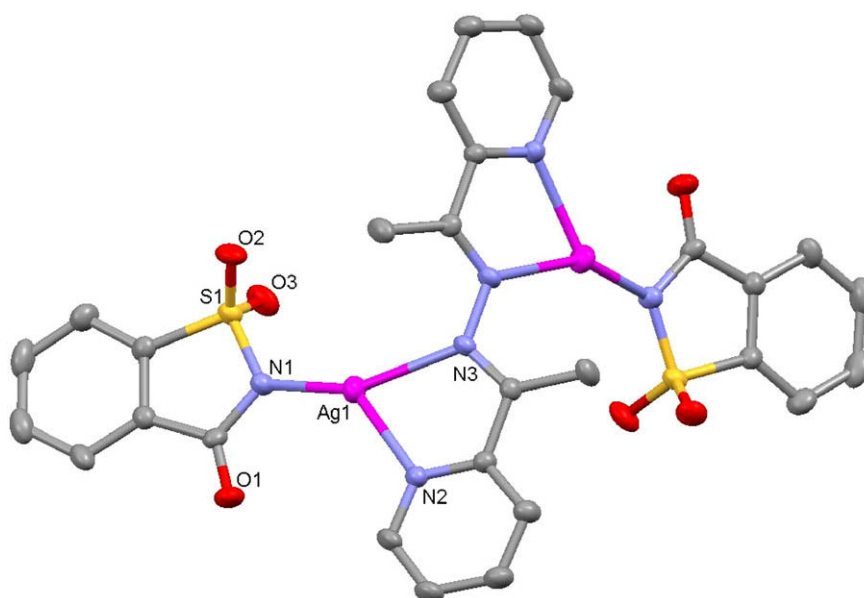


Fig. 1. A view of the binuclear structure of **1**. C–H hydrogen atoms were omitted for clarity.

molecules are connected by weak C–H...O hydrogen bonds into a three-dimensional network (Table 4).

The asymmetric unit of complex **2** is shown in Fig. 2 and selected bond lengths and angles are listed in Table 3. The structure of **2** is chiral ($P2_12_12_1$) and consists of one-dimensional helical chains, in which silver(I) centers are bridged by the bpma ligands as shown in Fig. 2. The bpma ligand acts as a tridentate ligand. Each silver(I) ion is coordinated in a distorted tetrahedral geometry by a

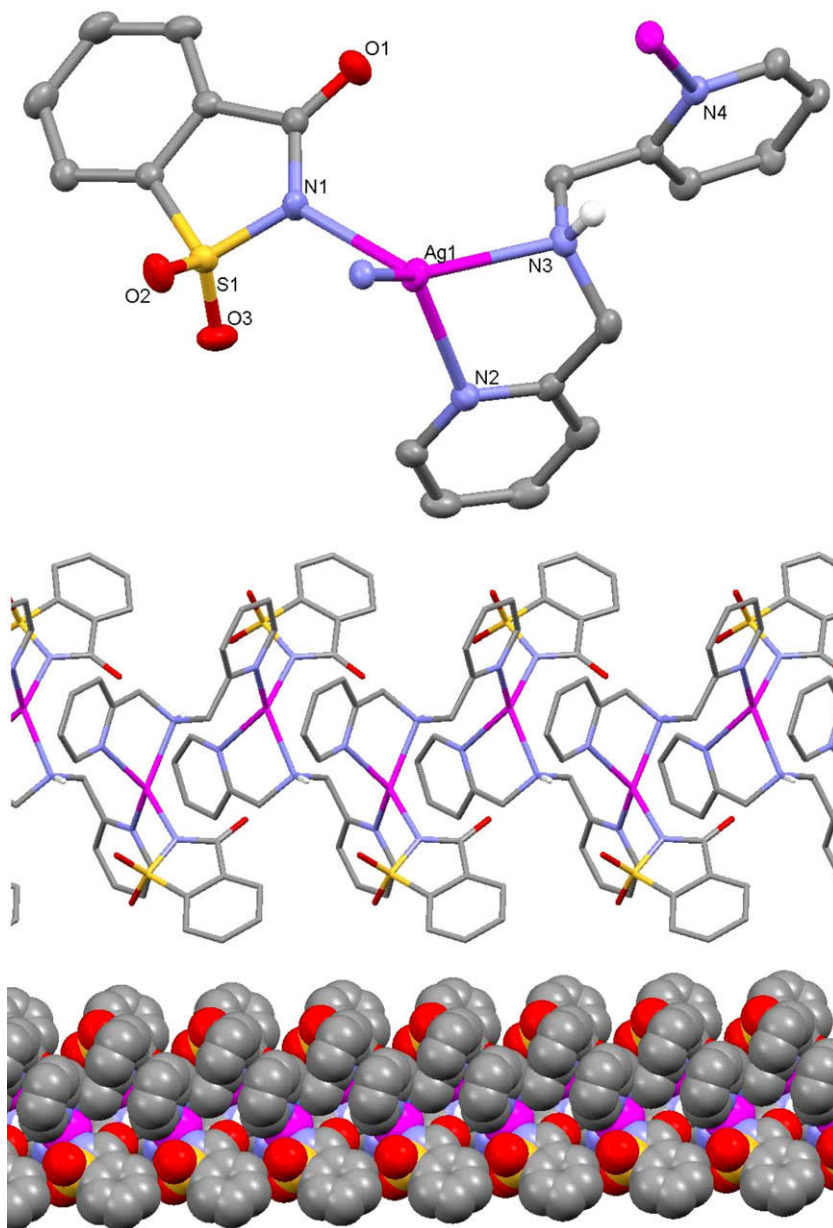


Fig. 2. Coordination environment around silver(I) and views of the one-dimensional chain in **2**. C–H hydrogen atoms were omitted for clarity.

monodentate (N) sac ligand, a bidentate (*N,N*) pyridylamine group of a bpma ligand and a py N atom of the symmetrically equivalent bpma ligand. As expected, the bpma ligand with the two py groups is essentially planar, possessing C_2 symmetry. Upon coordination, it is twisted around the amine N atom with torsion angles C12–C13–N3–C19 of $75.30(11)^\circ$ and C18–C19–N3–C13 of $61.65(12)^\circ$. Obviously, the twist of the ligand plays a crucial role in the generation of the molecular chirality [16–18]. The bpma ligand usually acts as a tripodal ligand [19–23], and as in the present case, its bridging behavior is rare. The Ag–N(bpma) bond distances are similar to those found in the mononuclear complexes [Ag(bpma)(PPh₃)]ClO₄ [21], [Ag(bpma)(P(*o*-tol)₃)]ClO₄ [20], and [Ag(bpma)(P(*o*-tol)₃)]NO₃ [23], whereas the Ag–N(sac) bond distance in complex **2** is slightly longer than that of complex **1**. The bpma ligand has an amine hydrogen atom, which is not involved in hydrogen bonding because of geometrical constraints. Therefore, the one-dimensional chains running along the axis *a* are linked into a three-dimensional network by weak C–H...O hydrogen bonds and C–H... π interactions as listed in Table 4.

3.3. Photoluminescence

The photoluminescent spectra of the ligands and their complexes have been measured at room temperature, and the emission spectra of the solid complexes are presented in Fig. 3. Na(sac) is emissive at 399 nm in the solid state when excited either at 275 or at 300 nm. Upon excitation at 385 nm, the emission spectrum of the bpeh ligand displays two main bands at 438 and 529 nm. Upon complexation, the emissions of complex **1** are not shifted compared to the free ligand, except for the decreased intensity of the band at 438 nm. Therefore, the metal-to-ligand charge transfer (MLCT) or metal centered (MC) $d \rightarrow s$ transitions are not possible due to the lack of appreciable red shifts and furthermore, the resemblance of the emission spectrum of **1** with that of bpeh indicates that the fluorescence of **1** is due to the ligand-based $\pi-\pi^*$ intraligand transitions at the excited state. On the other hand, the bpma ligand in the liquid form fluoresces at ca. 442 nm when excited at 350 nm, while the solid complex **2** features a distinct and broad emission band centered at 472 nm upon excitation at

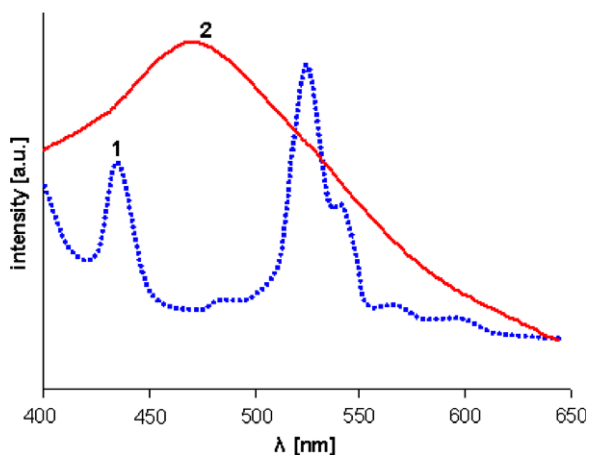


Fig. 3. Emission spectra of complex **1** (upon excitation at 385 nm) and complex **2** (upon excitation at 359 nm) in solid state at room temperature.

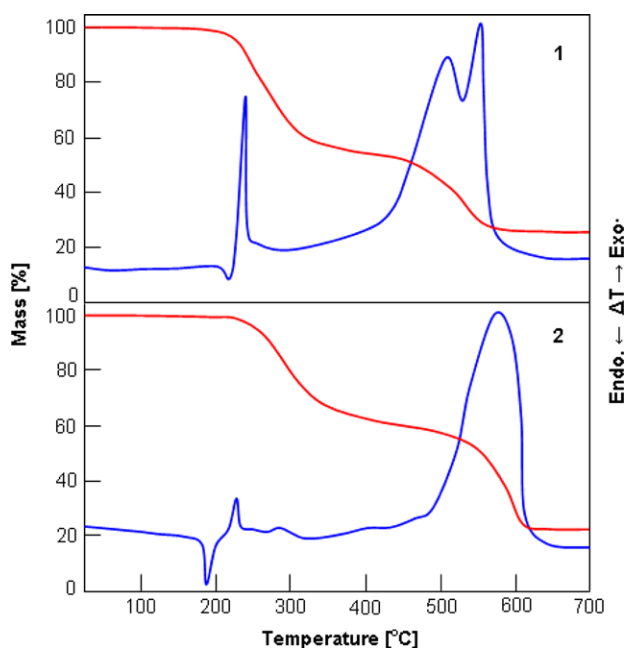


Fig. 4. DTA and TG curves of **1** and **2**.

359 nm. The observed red-shift of the emission maximum in **2** is probably due to a ligand-centered $\pi-\pi^*$ process including significant charge transfer character induced by the silver(I) ion. Commonly silver(I) complexes are weakly emissive at low temperatures [24,25] and the present results show that complexes **1** and **2** exhibit photoluminescence at ambient temperatures and their emissions are mainly originated from the co-ligands instead of sac.

3.4. Thermal decomposition

In order to characterize the complexes more fully in terms of thermal stability, their thermal behaviors were studied by TG and DTA at the atmosphere of air in the temperature range 25–900 °C. The TG and DTA curves are illustrated in Fig. 4. Both complexes are stable up to melting points (DTA peaks centered at 220 °C for **1** and 190 °C for **2**). After melting, complex **1** displays two-step decomposition between 195–361 °C and 362–577 °C with mass losses 43.4% and 29.8%. Although the mass loss in each stage is not consistent with individual mass of the bpeh and sac

ligands due to overlapping of decomposition reactions, the total mass loss (73.2%) at 577 °C agrees well with the calculated total mass of both ligands (73.6%). On the other hand, the DTA curves clearly indicate that an exothermic peak at 241 °C corresponds to the removal of bpeh, while highly exothermic peaks at 510 and 577 °C are characteristic for the decomposition of the sac moiety [26]. Complex **2** decomposes in two stages. The first stage in the temperature range 220–456 °C corresponds to the exothermic removal of the bpma ligand with a DTA peak at 288 °C (mass loss: found 40.4%, calcd. 40.7%). The degradation of the sac ligand takes place between 460 and 623 °C with a violently exothermic DTA peak centered at 581 °C (mass loss: found 37.5%, calcd. 37.2%). The decomposition of both complexes yields metallic silver as a decomposition product.

4. Conclusions

In conclusion, the use of two flexible achiral bis(pyridine) ligands as sources of conformational chirality leads to the formation of two chiral complexes $[\text{Ag}_2(\text{sac})_2(\text{bpeh})]$ (**1**) and $[\text{Ag}(\text{sac})(\text{bpma})]_n$ (**2**). Both complexes are characterized spectroscopically and crystallographically. The chirality of the molecular units is induced by the coordination of the ligands in a twisted chiral conformation. Weak, but significant intermolecular C–H \cdots O hydrogen bonds and C–H \cdots π interactions play very important role in the construction of three-dimensional supramolecular frameworks. Both complexes exhibit high thermal stabilities and are fluorescent at room temperature.

5. Supplementary material

CCDC 752881 and 752882 contain the supplementary crystallographic data for **1** and **2**. These data can be obtained free of charge via <http://www.ccdc.cam.ac.uk/conts/retrieving.html>, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk.

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