

两个基于氮、磷混合配体的银(I)配合物的合成、表征和荧光性质

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摘要: 在甲醇和二氯甲烷的混合溶液中合成了 2 个新的银(I)配合物, $[\text{Ag}_2(\text{dppp})_2(\text{phen})_2](\text{CF}_3\text{SO}_3)_2$ (**1**) 和 $[\text{Ag}_2(\text{dppm})_2(\text{dpq})_2](\text{CF}_3\text{SO}_3)_2 \cdot 3\text{CH}_3\text{OH}$ (**2**) (dppp=1,2-双(二苯膦)丙烷, dppm=1,2-双(二苯膦)甲烷, phen=1,10-菲咯啉, dpq=[2,3-*f*]吡嗪并[1,10]菲咯啉), 通过红外光谱、X 射线单晶衍射、核磁共振氢谱、荧光光谱和太赫兹时域光谱进行了分析和表征。**1** 是由 AgCF_3SO_3 , dppp 和 phen 以 1:1:1 的比例混合得到的双核化合物。中心原子 $\text{Ag}(\text{I})$ 通过双膦配体(dppp)桥联和含氮配体(phen)的螯合作用形成环。与 **1** 相似, **2** 是由 AgCF_3SO_3 和 dppm, dpq 以 1:1:1 的比例反应得到的相似的双核化合物。荧光光谱表明所有的发射峰均源于配体中的 $\pi-\pi^*$ 跃迁。

关键词: 银配合物; 荧光; 太赫兹谱

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Syntheses, Characterization and Luminescent Properties of Two Silver(I) Complexes Based on N-donor and P-donor Ligands

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Abstract: Two novel silver(I) complexes, $[\text{Ag}_2(\text{dppp})_2(\text{phen})_2](\text{CF}_3\text{SO}_3)_2$ (**1**) and $[\text{Ag}_2(\text{dppm})_2(\text{dpq})_2](\text{CF}_3\text{SO}_3)_2 \cdot 3\text{CH}_3\text{OH}$ (**2**) (dppp=bis(diphenylphosphino)propane, dppm=bis(diphenylphosphino)methane, phen=1,10-phenanthroline, dpq=pyrazino [2,3-*f*] [1,10]phenanthroline), have been synthesized in mixed solvent CH_3OH and CH_2Cl_2 and characterized by IR, single-crystal X-ray diffraction, ^1H NMR spectroscopy, fluorescence spectra and THz time domain spectroscopy (THz-TDS). Complex **1** is of a binuclear complex, which was generated by the reaction of AgCF_3SO_3 and dppp with phen in 1:1:1 molar ratio. In complex **1**, the central $\text{Ag}(\text{I})$ ion forms a ring by the bridging bisphosphine ligand (dppp) and chelating N-donor ligand (phen). Like **1**, **2** is of a binuclear complex, which was obtained by the reaction of AgCF_3SO_3 and dppm with dpq in 1:1:1 molar ratio. The luminescent spectra show that all these emissions are assigned to ligand centered $\pi-\pi^*$ transition. CCDC: 890658, **1**; 1571774, **2**.

Keywords: silver(I) complex; fluorescence; terahertz spectra

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So far, the structural diversity and potential applications of complexes had been studied in great detail^[1-5]. Complexes with coinage metals are applied to many areas and are widely served as functional materials, such as nanomaterials, magnetic materials, catalysts and antitumor drugs^[6-10].

Until now, a number of mixed-ligand Ag(I) diimine phosphine complexes such as $[\text{Ag}(\text{N-N})(\text{P-P})]^+$ system (P-P =various bis(phosphine) ligands) have been reported and their fluorescence properties have been investigated in various solvents and different temperatures^[11]. As we all known, silver(I) salts are classified as a kind of soft acid. According to the Hard-Soft-Acid-Base (HSAB) theory, as P-donor ligands, the rigid bis(diphenylphosphino)methane (dppm) and flexible bis(diphenylphosphino)propane (dppp) can easily coordinate with Ag(I) salts to help improve crystal quality. For the N-donor ligands, the chelating 1,10-phenanthroline ligand and one of its derivatives (dpq) were often used to synthesize complexes. In our previous reports, we have obtained some complexes with the novel structure and good fluorescence properties by the reaction of similar ligands and closed-shell d^{10} metal^[12-16].

In this paper, two novel silver(I) complexes, namely $[\text{Ag}_2(\text{dppp})_2(\text{phen})_2](\text{CF}_3\text{SO}_3)_2$ (**1**) and $[\text{Ag}_2(\text{dppm})_2(\text{dpq})_2](\text{CF}_3\text{SO}_3)_2 \cdot 3\text{CH}_3\text{OH}$ (**2**), where dppp=bis(diphenylphosphino)propane, dppm=bis(diphenylphosphino)methane, phen=1,10-phenanthroline, dpq=pyrazino[2,3-*f*][1,10]phenanthroline, have been synthesized and characterized by X-ray diffraction, IR, ^1H NMR, fluorescence

spectra and THz time domain spectroscopy (THz-TDS). The luminescent properties of these complexes are discussed.

1 Experimental

1.1 Materials and measurement

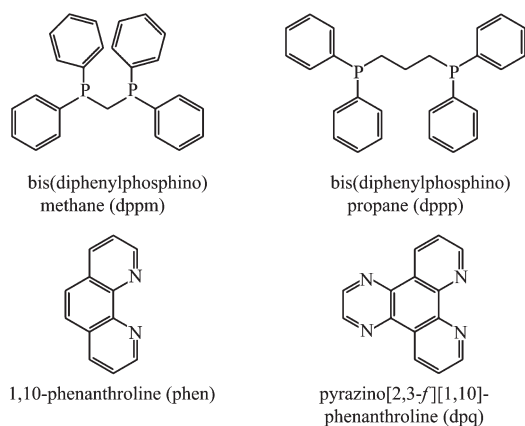
All chemical reagents are commercially available and used without furthermore treatment. FT-IR spectra (KBr pellets) were measured on a Perkin-Elmer Infrared spectrometer. C, H and N elemental analysis were carried out on an Elementar Vario MICRO CUBE (Germany) elemental analyzer. Room-temperature fluorescence spectra were measured on F-4500 FL Spectrophotometer. ^1H NMR was recorded at room temperature with a Bruker DPX 600 spectrometer. The THz absorption spectra were recorded on a THz time domain device of the Capital Normal University of China, carried out in a N_2 atmosphere to avoid the influence of water vapor, based on photoconductive switches for the generation and electro-optical crystal detection of the far-infrared light, effective frequency in the range of 0.2~2.8 THz^[17-18].

1.2 Synthesis of $[\text{Ag}_2(\text{dppp})_2(\text{phen})_2](\text{CF}_3\text{SO}_3)_2$ (**1**)

Complex **1** was prepared by the reaction of $\text{Ag}(\text{CF}_3\text{SO}_3)$ (0.051 4 g, 0.2 mmol), dppp (0.082 5 g, 0.2 mmol) and phen (0.036 0 g, 0.2 mmol) in the mixed solvents of 5 mL CH_2Cl_2 and 5 mL CH_3OH . The mixture was stirred for 6 hours and filtered. Colorless crystals were obtained from the filtrate after standing at room temperature for several days. Yield: 71%. Element analysis Calcd. for $\text{C}_{80}\text{H}_{68}\text{Ag}_2\text{F}_6\text{N}_4\text{O}_6\text{P}_4\text{S}_2$ (%): C, 56.55; H, 4.03; N, 3.30; Found(%): C, 56.27; H, 4.13; N, 3.28. IR data (cm^{-1} , KBr pellets): 3 439m, 3 056w, 1 621w, 1 588w, 1 570w, 1 510m, 1 485w, 1 436m, 1 422m, 1 264s, 1 224m, 1 151s, 1 100m, 1 032s, 999w, 841m, 758m, 745m, 730m, 695m, 664w, 638s, 572w, 525m, 482w, 439w. ^1H NMR (600 MHz, $\text{DMSO}-d_6$, 298 K): δ 7.1~8.6 (m, dppp-aromatic rings, phen-Heteroaromatic rings), 2.47 (t, dppp- CH_2)

1.3 Synthesis of $[\text{Ag}_2(\text{dppm})_2(\text{dpq})_2](\text{CF}_3\text{SO}_3)_2 \cdot 3\text{CH}_3\text{OH}$ (**2**)

Complex **2** was prepared in a manner similar to the described for **1**. A mixture of AgCF_3SO_3 (0.051 4 g,



Scheme 1 Structures of the ligands

0.2 mmol), dppm (0.076 9 g, 0.2 mmol) and dpq (0.046 4 g, 0.2 mmol) was dissolved in a mixture of 5 mL CH_2Cl_2 and 5 mL CH_3OH , stirred for 6 h and filtered. Yellow crystal was obtained from the filtrate after standing at the room temperature for several days. Yield: 57%. Element analysis Calcd. for $\text{C}_{80}\text{H}_{60}\text{Ag}_2\text{F}_6\text{N}_8\text{O}_6\text{P}_4\text{S}_2$ (%): C, 55.00; H, 3.46; N, 6.41. Found(%): C, 54.66; H, 3.53; N, 6.30. IR data (cm^{-1} , KBr pellets): 3 447m, 3 050m, 2 945w, 1 581m, 1 525w, 1 472m, 1 435m, 1 391m, 1 257s, 1 223m, 1 156s, 1 121m, 1 099m, 1 081m, 1 053w, 1 030s, 999m, 815m, 775m, 741s, 717m, 693s, 637s, 572m, 516m, 479m, 439m, 413w. ^1H NMR (600 MHz, $\text{DMSO}-d_6$, 298 K): δ 7.91~9.42 (m, overlap with the solvent peak signal, dpq-heteroaromatic rings, dppm-aromatic rings), 2.48 (s, dppm- CH_2)

1.4 Structure determination

Single crystals of the title complexes were mounted on a Bruker Smart 1000 CCD diffractometer equipped with a graphite-monochromated Mo $K\alpha$ ($\lambda =$

0.071 073 nm) radiation at 298 K. Semi-empirical absorption corrections were applied using SADABS program^[19]. All the structures were solved by direct methods using SHELXS program of the SHELXTL-97 package and refined with SHELXL-97^[20]. Metal atom centers were located from the E-maps and other non-hydrogen atoms were located in successive difference Fourier syntheses. The final refinements were performed by full matrix least-squares methods with anisotropic thermal parameters for non-hydrogen atoms on F^2 . The hydrogen atoms were generated geometrically and refined with displacement parameters riding on the concerned atoms.

Crystallographic data and experimental details for structural analysis are summarized in Table 1, and selected bond lengths and angles of complexes **1**~**2** are summarized in Table 2. The hydrogen bonds of complex **2** are listed in Table 3.

CCDC: 890658, **1**; 1571774, **2**.

Table 1 Crystallographic data for complexes **1**~**2**

Complex	1	2
Formula	$\text{C}_{80}\text{H}_{68}\text{Ag}_2\text{F}_6\text{N}_8\text{O}_6\text{P}_4\text{S}_2$	$\text{C}_{80}\text{H}_{60}\text{Ag}_2\text{F}_6\text{N}_8\text{O}_6\text{P}_4\text{S}_2$
Formula weight	1 699.12	1 747.16
Crystal system	Triclinic	Monoclinic
Space group	$P\bar{1}$	$C2/c$
Crystal size / mm	0.45×0.32×0.30	0.23×0.20×0.15
a / nm	1.306 90(12)	2.936 3(3)
b / nm	1.334 19(13)	1.356 25(11)
c / nm	1.353 01(15)	2.071 14(18)
α / (°)	97.879 0(10)	
β / (°)	104.509 0(10)	90.777 0(10)
γ / (°)	117.667(2)	
V / nm ³	1.932 4(3)	8.247 4(12)
Z	1	8
$F(000)$	864	3 752
Goodness-of-fit on F^2	1.040	1.055
R_{int}	0.034 8	0.105 8
$R_1 [I > 2\sigma(I)]^a$	0.052 2	0.060 2
$wR_2 [I > 2\sigma(I)]^b$	0.139 2	0.118 4
R_1 (all data) ^a	0.065 3	0.128 0
wR_2 (all data) ^b	0.153 4	0.129 7

$$^a R = \sum \|F_o\| - \|F_c\| / \sum \|F_o\|; ^b wR = [\sum w(|F_o|^2 - |F_c|^2)^2 / \sum w(F_o^2)]^{1/2}.$$

Table 2 Selected bond distances (nm) and bond angles (°) for complexes 1~2

Complex 1					
Ag(1)-P(1)	0.241 6(1)	Ag(1)-P(2)	0.245 3(1)	Ag(1)-N(1)	0.242 1(5)
Ag(1)-N(2)	0.244 5(4)				
P(1)-Ag(1)-N(1)	117.18(1)	P(1)-Ag(1)-N(2)	104.46(1)	N(1)-Ag(1)-N(2)	69.20(2)
P(1)-Ag(1)-P(2)	143.48(4)	N(1)-Ag(1)-P(2)	95.50(1)	N(2)-Ag(1)-P(2)	102.06(1)
Complex 2					
Ag(1)-P(1)	0.246 2(2)	Ag(1)-P(2)	0.242 9(2)	Ag(1)-N(1)	0.243 8(6)
Ag(1)-N(2)	0.244 2(6)				
P(2)-Ag(1)-N(1)	102.61(1)	P(2)-Ag(1)-N(2)	118.20(1)	N(1)-Ag(1)-N(2)	67.3(2)
P(2)-Ag(1)-P(1)	141.84(6)	N(1)-Ag(1)-P(1)	96.66(1)	N(2)-Ag(1)-P(1)	99.54(1)

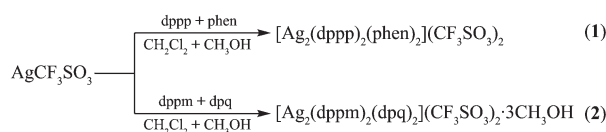
Table 3 Hydrogen bonds of complex 2

D-H...A	d(D-H) /nm	d(H...A) / nm	d(D...A) / nm	∠DHA / (°)
C(3)-H(3)···N(3)	0.093	0.251	0.281 9(1)	100

2 Results and discussion

2.1 Syntheses of the complexes

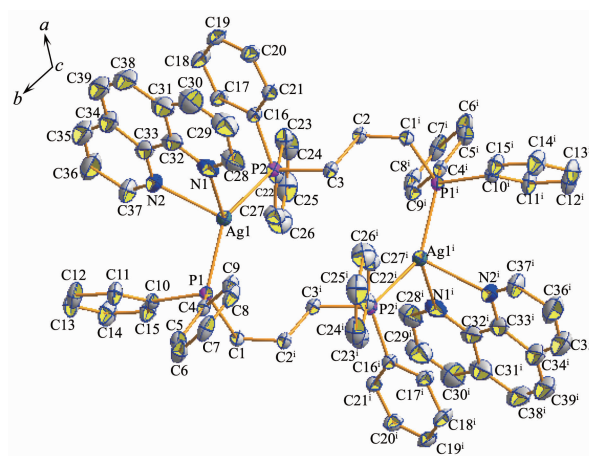
Functional Ag(I) complex **1** has been synthesized by one-pot reaction of silver(I) salts with dppp and phen ligands (Scheme 2). Complex **2** was prepared in a manner similar to the described for **1**, except for using dppm and dpq ligands. As is known to all, ligand is one of the factors that can influence the structures of the compounds. The dppp ligand has a long carbon chain and dppm ligand has an extremely strong rigid structure, so it's easy for the above two ligands to form a binuclear structure^[21-25] like complexes **1~2**. In complex **2**, there are hydrogen bonds and C-H... π intramolecular forces in molecular structure; however, there are no such forces in complex **1**.

Scheme 2 Synthetic routine for complexes **1** and **2**

2.2 Description of crystal structures

By Single-crystal X-ray diffraction analysis, complex **1** crystallizes in the triclinic crystal system with space group $P\bar{1}$. The asymmetric unit (Fig.1) is comprised of two Ag(I) ions, two dppp ligands and two 1,10-phenanthroline (phen) ligands, forming a

binuclear heteroleptic complex with a 0D coordination polymer. The Ag(I) ion adopts two types of four-coordinated modes, which are combined with two N atoms from phen ligand and two P atoms from dppp ligand establishing a distorted tetrahedral geometry for the metal. Ag-N (0.242 1(5), 0.244 5(4) nm) and Ag-P bond lengths (0.241 6(11), 0.245 3(11) nm) are typical Ag(I)-N and Ag(I)-P distances in coordination compounds^[26], respectively. Compared with the complex $[\text{Ag}(\text{dppp})_2](\text{NO}_3)_2$ ^[27], there are almost no so obvious differences in Ag-P bond distances (0.241 9(3),



All hydrogen atoms are omitted for clarity; Thermal ellipsoids drawn at the 30% probability level; Symmetry codes: ⁱ 1-x, 1-y, 1-z

Fig.1 Molecular structure of complex **1**

0.242 4(3) nm) and bond angle ($139.8(1)^\circ$) in complex **1**, implied that substituting anions have no effect on complex structure.

Unlike complex **1**, **2** crystallizes in the monoclinic crystal system with space group $C2/c$. Complex **2** is a binuclear heteroleptic complex formed with the same Ag(I), dppm and dpq ligands in 1:1:1 molar ratio. Each Ag atom is four-coordinated surrounded by two P atoms from dppm ligand and two N atoms from dpq ligand (Fig.2). The angles between Ag, N and P atoms are ranging from $67.3(2)^\circ$ to $141.84(6)^\circ$, indicating that the geometry around Ag atom is distortedly tetrahedral. Compared with complex $[Ag_2(\mu\text{-dppm})_2(\text{phen})_2](\text{NO}_3)_2$ ^[28], there are also no so obvious difference in bond distances (0.238 9(3), 0.240 6(9), 0.245 6(9), 0.246 1(3) nm) and bond angles ($68.9(9)^\circ \sim 44.5(3)^\circ$), since dpq ligand is the derivative of phen ligand. The C—H \cdots N intramolecular hydrogen bonds are observed (H \cdots N 0.251 nm, C—H \cdots N 100°). Moreover, the intramolecular C—H $\cdots\pi$ forces are also observed (H \cdots Cg 0.287 nm) in complex **2**, but in complex **1**, these two forces are not exist.

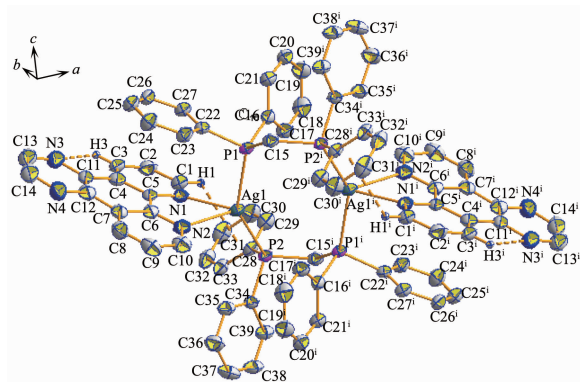


Fig.2 Molecular structure of complex **2**

2.3 Infrared spectroscopy and fluorescence spectra

The infrared spectra of **1** and **2** show C—C stretch vibration of the phenyl rings whose absorptions are found in 1 436 and 1 435 cm^{-1} . The middle absorptions around 3 056 and 3 050 cm^{-1} are caused by C—H vibration of the phenyl rings. The absorptions at 1 032 and 1 030 cm^{-1} are derived from trifluoromethane-

sulfonate (CF_3SO_3^-) in complexes **1** and **2**.

At room temperature, the solid-state excitation and emission spectra of complexes **1**~**2** and all the ligands were measured. When being excited at 322 nm, the dppm ligand displays a fluorescence emission peak at 431 nm, and the emission peak of the dppp ligand is found at 424 nm with excitation at 356 nm. It was found that the emission peak is centered at 525 nm with $\lambda_{\text{ex}}=386$ nm for complex **1**, and it is centered at 434 nm with $\lambda_{\text{ex}}=395$ nm for complex **2** (Fig.3). Why is there such a big difference in the emission of the two complexes? Firstly, there is no obvious difference on the part of diphosphine ligands. On the other hand, change of nitrogen ligand may be the major contributors to the distinct emission in complexes **1** and **2**. Compared with the dppp and dppm, complexes **1** and **2** all had different degrees of red-shift (101 and 10 nm, respectively). Moreover, complex **2** is red-shifted by about 17 nm compared with the dpq ligand which is excited at 381 nm and shows a fluorescence emission peak at 417 nm. These shifts of emission peak are derived from ligand-centered $\pi\text{-}\pi^*$ transition.

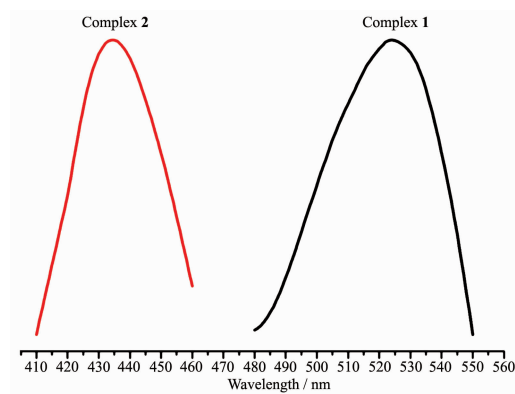


Fig.3 Luminescent spectra of **1** and **2** in the solid state at 298 K

Table 4 Fluorescent data of complexes **1** and **2** and the ligands

	$\lambda_{\text{ex}}/\text{nm}$	$\lambda_{\text{em}}/\text{nm}$
Complex 1	386	525
Complex 2	395	434
dppm	322	431
dppp	356	424
phen	373	419, 439
dpq	381	417

2.6 Terahertz (THz) absorption spectra

The room temperature terahertz (THz) absorption spectra of the AgCF_3SO_3 , dppm, dppp, phen, dpq and complexes **1**–**2** were measured in the range of 0.2~3.0 THz. All the above compounds have characteristic resonance peaks, which may be explained by the fact that in polar molecules the dipoles rotate and vibrate, resulting in strong absorption and chromatic dispersion. The peaks found for them are as follows: AgCF_3SO_3 0.69, 0.84, 1.00, 1.16, 1.30 THz; dppp 0.27, 0.37, 0.42, 0.56, 0.66, 1.07, 1.27, 1.38 THz; phen 1.05, 1.58, 1.76, 2.17, 2.51, 2.75 THz; dpq 0.64, 1.29, 1.70, 2.34, 2.69 THz; complex **1** 0.35, 0.53, 0.76, 0.94, 1.17, 1.53, 1.70, 1.87, 2.28, 2.52, 2.69 THz and complex **2** 0.24, 0.53, 0.76, 0.93, 1.67, 1.35, 1.52, 1.70, 1.88, 2.29, 2.46 THz (Fig.4). Comparing the THz absorption spectra of the products with those of the reactants, most peaks of the ligands and AgCF_3SO_3 disappeared or moved in the complexes. New peaks appear in the new complexes indicating that the THz absorption spectra are associated with the coordination of silver (I) ions and the ligands. Although the correspondence between the crystal structures and observed spectra does not allow a definitive characterization, it is possible to make tentative assignments of many of the observed features in the terahertz region for the samples. The results are a supplement to the THz spectroscopic properties of Ag complexes containing nitrogen and phosphorous ligands.

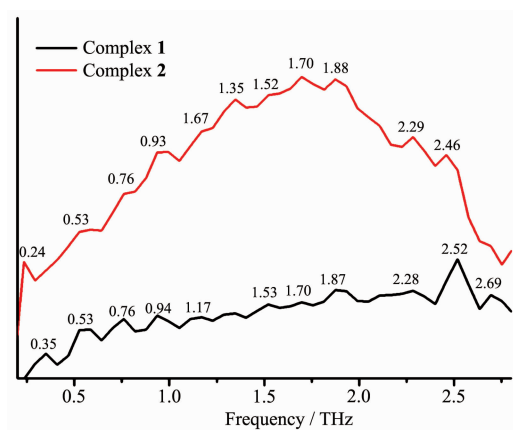


Fig.4 Terahertz spectra of complexes **1** and **2** in the range of 0.2~2.8 THz

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