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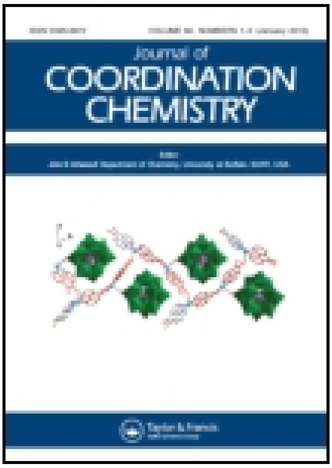

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A new isophorone-based ligand and its Ag(I) complex: crystal structures and luminescence

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Accepted author version posted online: 07 Apr 2014.Published online: 07 May 2014.

To cite this article: Feng Jin, Huizhi Zhu, Yan Yan, Hongping Zhou, Jieying Wu & Yupeng Tian (2014) A new isophorone-based ligand and its Ag(I) complex: crystal structures and luminescence, Journal of Coordination Chemistry, 67:7, 1198-1207, DOI: 10.1080/00958972.2014.912754

To link to this article: http://dx.doi.org/10.1080/00958972.2014.912754

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Journal of Coordination Chemistry, 2014
Vol. 67, No. 7, 1198–1207, http://dx.doi.org/10.1080/00958972.2014.912754

A new isophorone-based ligand and its Ag(I) complex: crystal structures and luminescence

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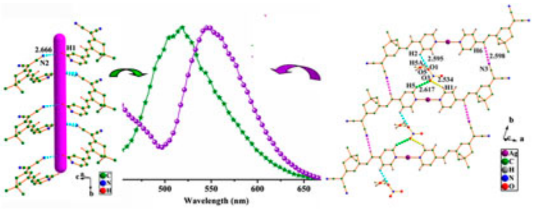
(*Received 19 December 2013; accepted 21 March 2014*)



The ligand in the Ag(I) complex shows more planar structure than the free ligand. Therefore, red shift emission and longer lifetime were realized by coordination of Ag(I) to the ligand.

An isophorone-based ligand with a delocalized π-electron system, 2-{5,5-dimethyl-3-[2-(pyridine-4-yl)ethenyl]cyclohex-2-enylidene}propanedinitrile (**L**), was synthesized. By assembling the ligand with $AgNO_3$, a mononuclear complex $[Ag(L)_2]NO_3 \cdot H_2O$ was obtained. Compared with the free ligand, the complex shows superior luminescent properties with large red-shift and longer fluorescence lifetime.

Keywords: Self-assembly; Ag(I) complex; Crystal structure; Luminescent property

1. Introduction

Synthesis of inorganic–organic hybrid complexes has become one of the most active fields in coordination chemistry and crystal engineering [1–7]. Simple association of ligand with metal salt through coordination and non-covalent interactions can assemble new coordination complexes which often exhibit better properties than the ligand [8]. A number of supramolecular complexes with specific topologies and superior properties have been synthesized by assembly of metal salts and organic ligands.

Coordination complexes based on Ag ion have attracted attention for structures, physicochemical properties, and potential applications as functional materials [9–17]. Synthesis of

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*Corresponding author. Email: zhouhongping@ahu.edu.cn suitable organic ligands is critical in construction of metal–organic coordination complexes. In order to construct supramolecular complexes with specific structures and properties, we have synthesized three multidentate ligands containing a π-conjugated system and pyridyl as well as dicyanoisophorone groups, 2-{5,5-dimethyl-3-[2-(pyridin-3-yl)-ethenyl]cyclohex-2enylidene}propanedinitrile (**L1**), 2-{5,5-dimethyl-3-[2-(pyridin-2-yl)-ethenyl]cyclohex-2enylidene}propanedinitrile (**L2**), and 2-{5,5-dimethyl-3-[2-(pyridine-4-yl)ethenyl]cyclohex-2enylidene}propanedinitrile (**L**). In the ligands, steric hindrance of the ring structure and dimethyl substituents of the isophorone unit is expected to hinder formation of intermolecular π···π interactions, which may avoid fluorescence quenching in the solid state [18]. The structures have been detailed in our previous report [13]. The only difference of the three ligands is that pyridine nitrogens are at different positions, displaying different orientations of the coordination sites, which may influence the coordination.

In our previous work, **L1** and **L2** have been used to construct Ag(I) coordination polymers, in which pyridine and nitrile are involved in coordination. Compared with the corresponding ligand, the Ag(I) complexes show superior luminescent properties [13]. To study the coordination ability and optical properties of the ligand caused by changing position of pyridine, reaction of 2-{5,5-dimethyl-3-[2-(pyridine-4-yl)ethenyl]cyclohex-2enylidene}propanedinitrile with $AgNO_3$ was carried out. Herein, we report the syntheses, crystal structures, and luminescence of **L** and $[Ag(L)_2]NO_3 \cdot H_2O$.

2. Experimental

2.1. *Materials and physical measurements*

All commercially available chemicals are of analytical grade and used without purification. Elemental analyses were carried out on a Perkin-Elmer 240 analyzer. ^1H and ^{13}C NMR spectra were recorded on a Bruker AV 400 spectrometer using $CDCl_3$ and $(CD_3)_2SO$ as solvents. The mass spectra were obtained on a Micromass GCT-MS spectrometer. IR spectra were recorded as KBr disks from 4000 to 400 cm^{-1} on a Nicolet Nexus 870 spectrophotometer. Solid state luminescence spectra were measured on a F-4500 FL spectrophotometer. In the measurements of emission and excitation spectra, the pass width is 5 nm. For time-resolved fluorescence measurements, the fluorescence signals were collimated and focused onto the entrance slit of a monochromator with the output plane equipped with a photomultiplier tube (HORIBA HuoroMax-4P). The decays were analyzed by least-squares. The quality of the exponential fits was evaluated by the goodness of fit (χ^2).

2.2. *Preparation of L and the complex*

L: 2-(3,5,5-Trimethylcyclohex-2-enylidene)malononitrile (2.60 g, 14 mM), CH_3CN (100 mL), pyridine-4-carboxaldehyde (1.50 g, 14 mM), and piperidine (2 mL) were added to a round-bottom flask. The reaction mixture was stirred for 12 h at 40 °C. After cooling to room temperature, yellow crystals formed slowly. The product was filtered and recrystallized from MeOH. Yield: 2.90 g (75%). Anal. Calcd for $C_{18}H_{17}N_3$: C, 78.45; H, 6.17; N, 15.25. Found (%): C, 78.28; H, 6.41; N, 15.56. ^1H NMR (400 MHz, $CDCl_3$): δ 1.10 (s, 6H, –CH_3), 2.48 (s, 2H, –CH_2), 2.63 (s, 2H, –CH_2), 6.95 (d, $J = 16$, 2H, alkene-CH), 7.13 (s, 1H, alkene-CH), 7.36 (d, $J = 6$, 2H, Ar–H), 8.65 (d, $J = 6$, 2H, Ar–H); ^{13}C NMR (100 MHz, CD_3Cl): δ 28.0 (12), 32.0 (11, 13), 39.1 (14), 42.9 (10), 80.6 (16), 112.2 (C17, C18), 113.0 (C3, C4), 121.3 (C9), 133.2 (C6), 133.7 (C7), 142.8 (C5), 150.5 (C1, C2), 168.9 (C15) [The serial number of the C atoms are shown in figure 1(a)]. IR (KBr, cm^{-1}): 3115, 2931 *v*(m, Ar–H), 2868, 2596 *v*(s, $-CH_3$), 2219 *v*(s, C≡N), 1567, 1526 *v*(s, C=C); MS (EI) (*m/z*): Calcd for $C_{18}H_{17}N_3$: 276.15 $[HM]^+$; Found: 276.06 $[HM]^+$.

$[Ag(L)_2]NO_3 \cdot H_2O$: A clear methanol solution (10 mL) of $AgNO_3$ (0.169 g, 1 mM) was carefully layered onto a solution of **L** (0.552 g, 2 mM) in dichloromethane (15 mL). Yellow needle crystals suitable for single-crystal X-ray diffraction were obtained by slow interlayer diffusion in the dark. Yield: 0.49 g (66%). Anal. Calcd for $C_{36}H_{36}AgN_7O_4$: C, 58.54; H, 4.91; N, 13.27. Found (%): C, 58.26; H, 4.61; N, 13.59. ^1H NMR (400 MHz, DMSO-d_6): δ 1.03 (s, 6H, $-CH_3$), 2.55 (s, 2H, $-CH_2$), 2.65 (s, 2H, $-CH_2$), 7.01 (s, 1H, alkene-CH), 7.30, 7.26 (d, $J = 16$, 2H, –CH = CH–), 7.72–7.78 (s, 3H, alkene-CH, Ar–H), 8.66 (d, $J = 4$, 2H, Ar–H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 27.4 (C13), 31.6 (C14, C15), 38.0 (C12), 42.4

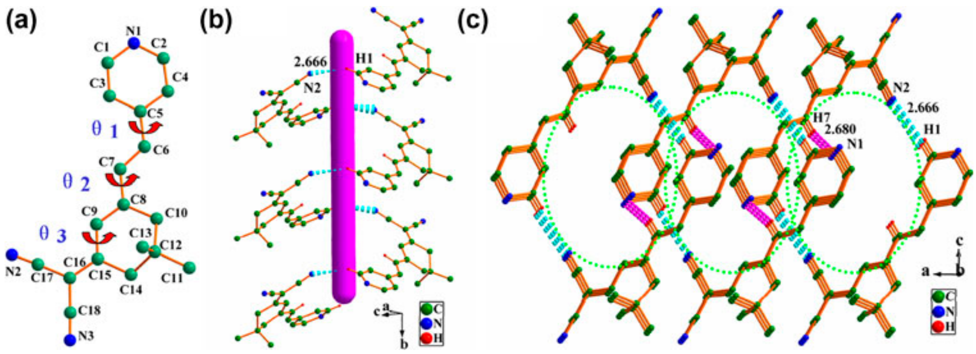


Figure 1. (a) The molecular structure of **L**. Hydrogens are omitted for clarity. (b) The 1-D helical structure of **L** showing the C–H···N hydrogen bonding interaction. (c) The 2-D structure of **L**. The dotted lines represent weak interactions. Hydrogens not participating in hydrogen bonding are omitted for clarity.

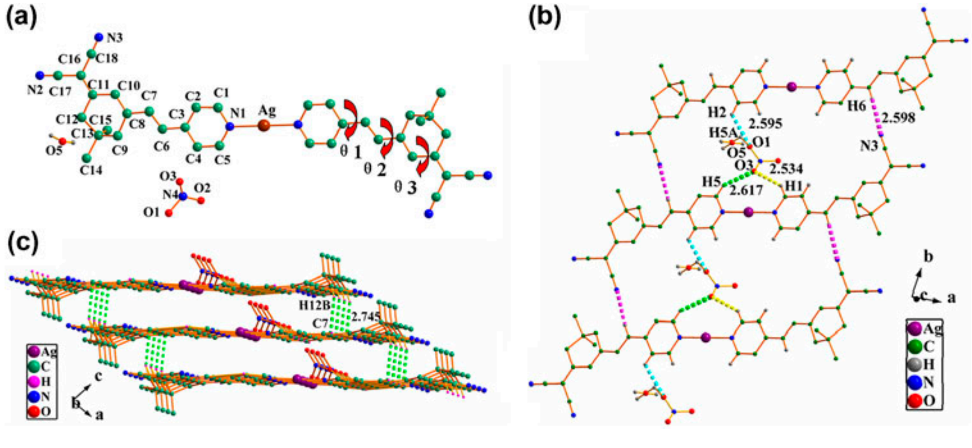


Figure 2. (a) The molecular structure of the complex. Hydrogens are omitted for clarity. (b) The 1-D structure of the complex. (c) The 2-D structure of the complex. The dotted lines represent weak interactions. Hydrogens not participating in hydrogen bonding and water molecules are omitted for clarity.

(C9), 78.6 (C16), 112.7 (C17, C18), 113.4 (C2, C4), 122.2 (C18), 133.5 (C6), 134.9 (C7), 144.7 (C3), 149.9 (C1, C5), 170.1 (C11) [The serial number of the C atoms are shown in figure 2(a)]. IR (KBr, cm^{-1}): 3516 ν(s, O–H), 3117, 2938 ν(s, Ar–H), 2871, 2592 ν(s, $-CH_3$), 2221 ν(s, C≡N), 1565, 1527 ν(s, C=C), 1427 ν(s), 1337 ν(s, NO_3^-); MS (EI) (*m/z*): Calcd for $C_{36}H_{34}AgN_6$: 657.19 $[AgL_2]^+$; Found: 657.01 $[AgL_2]^+$; $C_{18}H_{17}AgN_3$: 382.05 $[AgL]^+$; Found: 382.12 $[AgL]^+$.

2.3. *Crystallographic measurements*

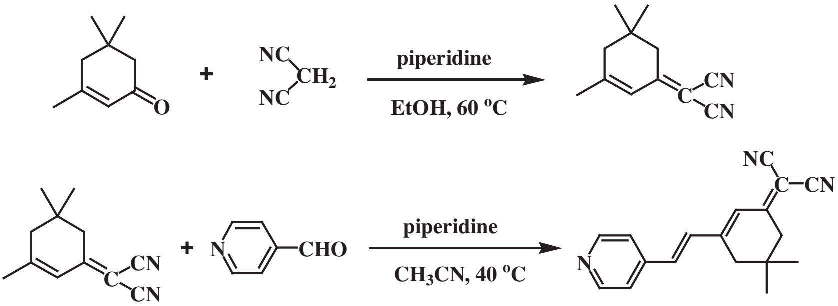
X-ray diffraction measurements were performed on a Bruker SMART CCD area detector using graphite monochromated Mo Kα radiation ($\lambda = 0.71069$ Å) at 298(2) K. Intensity data were collected in the variable ω-scan mode. The structures were solved by direct methods and difference Fourier syntheses. The non-hydrogen atoms were refined anisotropically and hydrogens were introduced geometrically. Calculations were performed with SHELXTL-97 program package [19].

3. Results and discussion

3.1. *Syntheses and characterization*

The synthetic procedure of the ligand is shown in scheme 1. It is a multidentate asymmetric ligand and contains three coordination sites, one pyridine and two nitrile nitrogens, which may completely or partially coordinate with Ag ion to construct different complexes. Single crystals of the complex were obtained in methanol and dichloromethane mixed solvent system by reaction of the ligand with $AgNO_3$ at room temperature. The single crystal is air stable and photostable and can retain its structural integrity at room temperature for a few months.

The ^{1}H NMR spectrum of ligand agrees with its structure and displays 17 proton resonances (figure S1, see online supplemental material at http://dx.doi.org/10.1080/00958972.2014.912754). The double peaks at δ H 8.65 and 7.36 can be assigned to four hydrogens of pyridyl. Peaks at δ H 6.95 are assigned to the vinyl-H based on the 16 Hz coupling constant. The remaining resonances can be assigned easily. ^{1}H NMR spectra of complex are different from that of the free ligand, as shown in figure S3 of Supplemental material. In the complex, the resonances of the protons of the conjugated backbone appear downfield relative to the corresponding protons of free ligand caused by the electron-withdrawing effects of metal [20].



Scheme 1. Synthetic route to **L**.

In the IR spectrum of the complex, broad band at 3516 cm^{-1} indicates O–H stretch of water. The intense ν(C≡N) of nitrile are identified at 2219 cm^{-1} for **L** and 2221 cm^{-1} for the complex. The small difference of ν(C≡N) between the complex and the free ligand suggests the non-involvement of nitrile groups in coordination. The facts agree well with the results determined by X-ray single-crystal diffraction.

3.2. *Description of X-ray crystal structures*

Details of the crystal parameters, data collections, and refinements are listed in table 1, and selected bond lengths and angles are given in table 2. As shown in figure 1(a), the conjugated system of the free ligand with N1, C1, C2, C3, C4, C5, C6, C7, C8, C9, C15, and

Table 1. Crystal data and refinements of the ligand and the Ag(I) complex.

Compound	**L**	Complex
Empirical formula	$C_{18}H_{17}N_3$	$C_{36}H_{36}AgN_7O_4$
Formula weight	275.35	738.59
Crystal system	Monoclinic	Triclinic
Space group	$P2_1/n$	$P\bar{1}$
a [Å]	6.045(5)	5.515(5)
b [Å]	7.274(5)	9.326(5)
c [Å]	35.999(5)	18.453(5)
α [°]		89.933(5)
β [°]	91.014(5)	82.129(5)
γ [°]		81.772(5)
V [Å^3]	1583(2)	930(1)
Z	4	1
T [K]	298(2)	298(2)
D_{Calcd} [g cm^{-3}]	1.156	1.318
μ [mm^{-1}]	0.070	0.587
θ Range [°]	1.13–25.00	1.11–25.00
Total no. data	10,825	6613
No. unique data	2776	3230
No. params. refined	192	240
R_1	0.0498	0.0582
wR_2	0.1368	0.1915
GOF on F^2	1.064	1.029

Table 2. Selected bond lengths (Å) of the ligand and the Ag(I) complex.

L			
N(1)–C(2)	1.337(3)	N(1)–C(1)	1.339(3)
C(8)–C(9)	1.358(3)	C(8)–C(7)	1.444(2)
C(6)–C(7)	1.337(3)	C(6)–C(5)	1.460(3)
C(5)–C(4)	1.398(3)	C(1)–C(3)	1.370(3)
C(5)–C(3)	1.391(3)	C(4)–C(2)	1.371(3)
C(16)–C(17)	1.431(3)	C(16)–C(18)	1.429(3)
Complex			
Ag–N(1)	2.137(4)	N(1)–C(1)	1.333(7)
N(1)–C(5)	1.320(8)	C(3)–C(2)	1.385(7)
C(3)–C(4)	1.382(7)	C(6)–C(3)	1.456(6)
C(6)–C(7)	1.338(6)	C(8)–C(7)	1.456(6)
C(11)–C(16)	1.372(6)	C(11)–C(10)	1.436(6)
C(10)–C(8)	1.354(6)		

C16 form a mean plane. The deviation to the mean plane is 0.087 Å, and the torsion angles between the pyridine ring and its neighboring double bond as well as the adjacent double bonds are 80.79° for $\theta 1$, 8.21° for $\theta 2$, and 3.01° for $\theta 3$. However, in the complex [figure 2(a)], the corresponding deviation to the mean plane is 0.036 Å, and the torsion angles are 85.93° for $\theta 1$, 6.43° for $\theta 2$, and 1.64° for $\theta 3$. The data obviously indicate better planarity of the conjugated backbone of the ligand in the complex.

As demonstrated in figure 1(b), adjacent molecules are linked by C(1)–H(1)···N(2) hydrogen bonding interactions to form a chain along the *b* axis with H(1)···N(2) and C(1)···N(2) distances of 2.666(2) and 3.504(3) Å, respectively. The chains are connected through C(7)–H(7)···N(1) hydrogen bonding interaction to generate the 2-D structure. The H(7)···N(1) and C(7)···N(1) distances are 2.680(5) and 3.548(3) Å, respectively. The C(7)–H(7)···N(1) angle is 155.659(1)°.

As shown in figure 2(a), the Ag(I) complex is mononuclear, and the Ag(I) shows linear N_2-coordination geometry by two pyridine nitrogens from two ligands, without showing any nitrile-silver(I) interactions, different from the reported complexes $\{[Ag(L1)(NO_3)]\cdot 0.5C_6H_6\}_n$ and $\{[Ag(L2)(NO_3)]\cdot 0.5C_6H_6\}_n$ [13]. The Ag–N(1) bond distance (2.137 (4) Å) is comparable to those of similar N–Ag complexes [21]. The angle between the coordinating donors may influence the coordination mode of the ligand. In the ligand, the pyridine nitrogen and the nitrile nitrogen are at opposite ends of the molecule, which, combined with the rigid backbone, would make it difficult for the pyridine and nitrile nitrogen to involve in coordination simultaneously [22]. Therefore, the ligand is not suitable for constructing helical or other coordination polymers [23–26]. The complex cation is centrosymmetric and charge is balanced by the uncoordinated nitrate. A water molecule is located in the asymmetric unit and resides on an inversion center.

The nitrile groups are not involved in coordination. However, the C–H···N hydrogen bonding interaction based on a nitrile is formed and plays an important role in formation of higher dimensional structure through C–H···N hydrogen bonding interactions. As shown in figure 2(b), neighboring molecules are linked through C(6)–H(6)···N(3) hydrogen bonding interactions to form a 1-D structure with H(6)···N(3) and C(6)···N(3) distance of 2.598(6) and 3.510(8) Å, respectively. The C(6)–H(6)···N(3) angle is 166.988(3)°. The 1-D structures are connected through weak C(12B)–H(12B)···π interactions with H(12B)···C(7) distance of 2.745(4) Å to generate a 2-D structure, as displayed in figure 2(c). Multiple hydrogen bonding interactions based on nitrates and waters provide further stability to the 1-D and 2-D structures. As expected, because of the steric hindrance of the ring structure and the dimethyl substituents of isophorone, no π···π interactions were observed in the free ligand or complex [13], which may have significant influence on the solid-state emission of the crystals [27–32].

3.3. *Time-dependent density functional theory (TDDFT) calculations*

To further investigate the relation between molecular structures and optical properties, quantum chemical calculations based on time-dependent density functional theory (TDDFT) were carried out using the Gaussian 03 program. The B3LYP/6-31G basis sets were used for calculations. The electron-cloud distribution of frontier molecular orbitals is shown in figure 3. The electron cloud is delocalized throughout the backbone of the ligand. The HOMO and LUMO diagrams of the ligand and the complex both clearly show the *n*–π* and π–π* transitions upon photoexcitation.

3.4. *Luminescent properties*

Photoluminescence of Ag(I) coordination complexes has been extensively studied due to their potential applications as luminescent materials. The emission bands of Ag(I) complexes usually originate from π–π^* intra-ligand, or ligand-to-metal charge transfer transitions [10, 33–35]. Many Ag(I) complexes show excellent optical properties [13, 22, 36]. Therefore, for potential applications as luminescent materials, the luminescent properties of **L** and the complex have been investigated in the solid state at room temperature with excitation wavelength at 370 nm. Figure 4 presents the comparison of the emission spectra. **L** exhibits emission maximum at 519 nm, assigned to the n–π^* transition of the nitrile and the π–π^* transition of the backbone. Compared to that of **L**, the emission spectrum of the complex is red-shifted to 546 nm and also from the n–π^* and π–π^* transitions which can be confirmed by the quantum chemical calculations. The structure analysis indicates that

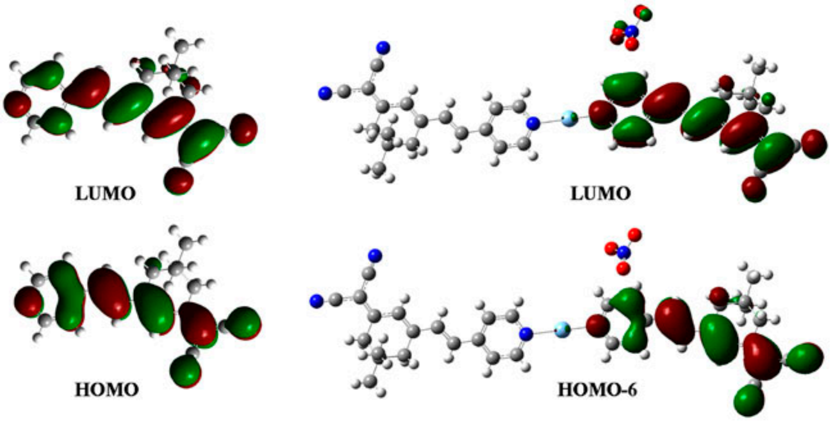


Figure 3. The electron-cloud distribution of frontier molecular orbitals of **L** and the Ag(I) complex.

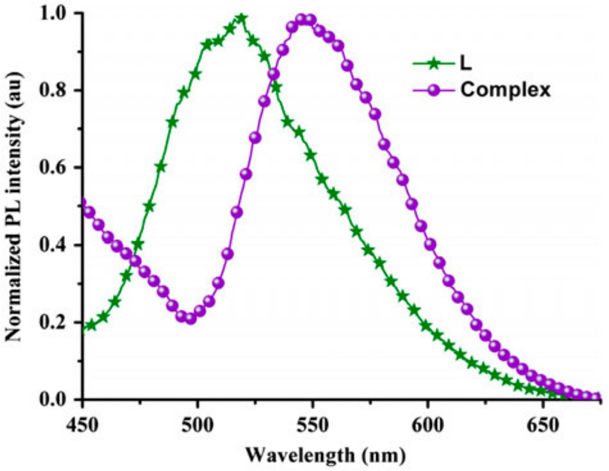


Figure 4. Normalized solid-state emission spectra of **L** and the Ag(I) complex at room temperature.

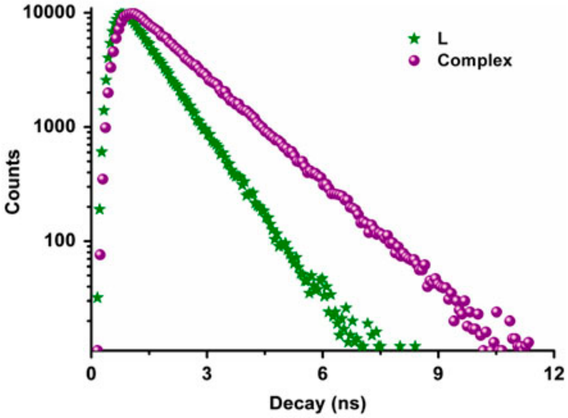


Figure 5. Time-resolved fluorescence curves of **L** and the Ag(I) complex in the solid state at room temperature.

incorporation of Ag(I) effectively increases the conformational rigidity of the ligand, which would lead to a smaller HOMO–LUMO energy gap [37] and reduce the non-radiative decay of the intraligand (π–π*) excited state [38].

To further understand the fluorescent properties, the fluorescence decay profiles of **L** and the complex were measured at their optimal excitation wavelengths in the solid state at room temperature. The results are shown in figure 5. **L** has very short fluorescence lifetime of 0.10 ns. However, the fluorescence lifetime of the complex is 0.39 ns. The luminescent properties indicate that the emission spectrum of the organic compound is remarkably influenced by its incorporation into $AgNO_3$.

4. Conclusion

A new ligand containing pyridine and nitrile, 2-{5,5-dimethyl-3-[2-(pyridin-4-yl)ethenyl]cyclohex-2-enylidene}propanedinitrile, has been synthesized and used to construct a mononuclear Ag(I) complex. Only a monodentate bonding through the pyridine nitrogen is observed for the ligand, different from the reported **L1** and **L2**. The changes of the donor positions have a major influence on the structures of the complexes. The crystal structure analyses indicate that the ligand in the complex shows more planar structure than the free ligand. The superior luminescent properties with red-shift emission and longer fluorescence lifetime are realized by coordination of Ag ion to the ligand, indicating that coordination is a suitable way to synthesize new fluorescent materials.

Supplementary material

Crystallographic data for the compounds have been deposited with the Cambridge Crystallographic Data Center; CCDC reference number is 914586 for **L** and 952155 for $[Ag(L)_2]NO_3 \cdot H_2O$. Copies of the information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Tel: +(44) 01223 762910; Fax: +(44) 01223336033; E-mail: deposit@ccdc.cam.ac.uk or visit website: http://www.ccdc.cam.ac.uk/deposit).

Funding

This work was supported by Program for New Century Excellent Talents in University (China); Doctoral Program Foundation of Ministry of Education of China [grant number 20113401], [grant number 110004]; National Natural Science Foundation of China [grant number 21271003], [grant number 21271004]; the 211 Project of Anhui University, the Anhui University Students Research Training Program [grant number kyxl2012020]; the Graduate Students Innovative Program of Anhui University [grant number 10117700063]; and Natural Science Foundation of Education Committee of Anhui Province [grant number KJ2012A024], [grant number KJ2013B201].

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